UNITED STATES DISTRICT COURT
DISTRICT OF NEW JERSEY

THERAVANCE BIOPHARMA R&D IP, LLC, et al., Plaintiffs, v. EUGIA PHARMA SPECIALITIES LTD., et al., Defendants.	C.A. No. 1:23-cv-00926-KMW-AMD (consolidated)

DECLARATION OF DR. MICHAEL ZAWOROTKO

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I. INTRODUCTION

1. I have been asked by Defendants Mankind Pharma Ltd. and Lifestar Pharma LLC (collectively, "Mankind") and Cipla Limited and Cipla USA, Inc. (collectively, "Cipla") (Mankind and Cipla, collectively "Defendants") to provide this declaration concerning the meaning of certain claim terms in U.S. Patent Nos. 8,541,451 (the "'451 patent") (Ex. A), 9,765,028 (the "'028 patent") (Ex. G), 10,100,013 (the "'013 patent") (Ex. O), 10,550,081 (the "'081 patent") (Ex. N), 11,008,289 (the "'289 patent") (Ex. I), 11,649,209 (the "'209 patent") (Ex. K), 11,691,948 (the "'948 patent") (Ex. C), and 11,858,898 (the "'898 patent") (Ex. E) (collectively, "the Polymorph Patents"). I understand the following claims of the Polymorph Patents have been asserted against Defendants:

Patent No.	**Asserted Claims**
8,541,451	1-8, 12
9,765,028	1-6
10,100,013	1-9
10,550,081	1-4
11,008,289	1-9
11,649,209	1-11
11,691,948	1-21
11,858,898	1-27

2. I have provided technical background related to the Polymorph Patents and crystalline solids generally, my opinions about the definition of the person of ordinary skill in the art ("POSA") in July 2009, and the meaning of the disputed claim terms to a POSA in July 2009. If asked, I am prepared to testify regarding the matters I discuss in this declaration.

3. I understand that Plaintiffs Theravance Biopharma R&D IP, LLC, Theravance Biopharma Ireland Limited, Theravance Biopharma US, Inc., Mylan Ireland Limited, and Mylan

Specialty L.P. (collectively, "Plaintiffs") have filed suit against Defendants Mankind and Cipla. For ease of reference, I will refer to Cipla and Mankind together as "Defendants," and I will refer to the Plaintiffs and Defendants together as "the Parties."

4. I understand that the Parties filed a Joint Claim Construction Statement on February 11, 2025, providing proposed constructions for certain claim terms of the Polymorph Patents.

5. I have considered and relied on the Polymorph Patents and their prosecution histories, the Joint Claim Construction Statement, and the documents identified in Appendix 1, from the perspective of a POSA as of July 2009. I have also relied on my knowledge, education, and professional expertise of over 42 years in the field of crystallography (the science of crystalline solids and their design through crystal engineering) and its applications (study of the properties of crystalline solids in the context of pharmaceutical science and industrial separations). I understand that there are other patents asserted against Defendants in this case, and I am not offering any opinions on any patents other than the Polymorph Patents.

6. I am being compensated for my work in this case at the rate of $600 per hour plus expenses. My compensation is in no way tied to the outcome of this case or to the content of this declaration.

II. BACKGROUND AND QUALIFICATIONS

7. I am qualified to render opinions regarding the subject matter of the Polymorph Patents. I have over 42 years of professional experience relating to this subject matter. Below I have summarized my relevant education and experience.

8. I currently serve as Bernal Chair of Crystal Engineering and Science Foundation of Ireland Research Professor in the Department of Chemical Sciences at the University of Limerick, Limerick, Ireland. I have held this position since November 2013. From June 2017 through September 2022, I also served as Co-Director of the Synthesis and Solid State Pharmaceutical

Centre, SSPC, hosted by the University of Limerick. SSPC is a global hub for the pharmaceutical industry that is supported by > €60 million of government and industrial funding from 2019-2025.

9. I received my Bachelor of Science in Chemistry from Imperial College (London, U.K.) in June 1977. I obtained my Ph.D. in Chemistry from the University of Alabama in August 1982. My Ph.D. research was conducted under the supervision of Professor Jerry L. Atwood and focused upon synthesis and structural chemistry of organic, organometallic, and coordination compounds. I served as a post-doctoral fellow at the University of Victoria, Canada, from April 1982 until August 1985. My postdoctoral research concerned synthesis and structural chemistry of coordination and organometallic compounds.

10. From August 1985 to January 1998, I served on the faculty of Saint Mary's University (Halifax, Nova Scotia), where I held various positions including Chair of the Department of Chemistry (August 1993 to August 1997).

11. From January 1998 to September 1999, I served as Professor of Chemistry and Dean of the College of Arts and Sciences at the University of Winnipeg, Manitoba.

12. I served as a Professor in the Department of Chemistry at the University of South Florida in Tampa (the ninth largest university in the United States at the time) from September 1999 until December 19, 2013. During my tenure at the University of South Florida, I served as Chair of the Department of Chemistry from September 1999 until August 2008, and I was appointed to the position of Distinguished Professor in 2013.

13. I have taught courses on general chemistry, inorganic chemistry, organometallic chemistry, crystal engineering, and solid-state chemistry, including testing methods used to identify and characterize the properties of solids.

14. I was awarded the President's Award for Research Excellence (Saint Mary's University, 1994), am an Honorary Member of the Academy of Science of Higher School of Ukraine, have been awarded Visiting Professorships to Université Louis Pasteur (Strasbourg, France, 1999) and the Institute of Biology and Chemistry of Proteins (Lyon, France, 2001). In 2012, I was appointed Fellow of the American Association for the Advancement of Science, and in 2015, I was admitted as a Fellow of the Royal Society of Chemistry and as a Fellow of the Institute of Chemistry of Ireland. In 2017, I was admitted as a Fellow of the Learned Society of Wales and I was awarded the highest honor granted by the Science Foundation of Ireland (SFI), "SFI Researcher of the Year." In 2019, I became a member of the Royal Irish Academy.

15. I served as Founding Editor of *Crystal Engineering*, published by Elsevier from 1997-2003. I served as Associate Editor of the journal *Crystal Growth and Design*, a publication by the American Chemical Society, from 2007-2019. I currently serve on the editorial boards of two journals in the field of crystallography: *Journal of Chemical Crystallography* and *IUCr Journal.*

16. I co-organized the first international meeting devoted to the subject of crystal engineering (NATO-ASI meeting from 09/09/1996 to 09/20/1996). I served as Co-Vice-Chair of the Second Gordon Conference on Crystal Engineering that was held in June 2012. I served as Co-Chair of the Third Conference on Crystal Engineering from June 1-6th 2014.

17. I co-edited the proceedings of the aforementioned NATO-ASI meeting (Seddon, K.R. and Zaworotko, M.J. "Crystal Engineering: the Design and Application of Functional Solids," Kluwer, 1999). I co-edited a book titled "Organic Crystal Engineering: Frontiers in Crystal Engineering," that was published by Wiley in 2010.

18. I have supervised the Ph.D. dissertations of approximately forty (40) students and supervised the research of approximately fifty (50) postdoctoral research associates. I currently supervise a research group consisting of five (5) Ph.D. students, one (1) visiting scientist and five (5) postdoctoral research associates.

19. I have published approximately 520 peer-reviewed articles in academic journals, and I have twenty-six (26) issued patents where I am a named inventor. Most of the publications deal generally with crystallography including crystallization, X-ray crystallography, crystal engineering, crystal packing, polymorphism, or related subjects. Nine (9) of the patents have been licensed. In addition, multiple of my patents are pending, including five (5) filed since I joined the University of Limerick in November 2013.

20. I have published more than 1200 crystal structures, many of which address the structure and properties of molecular solids, including hydrates and solvates of drug molecules or drug candidate molecules.

21. During the course of my academic research, I have designed novel drug substances (also known as active pharmaceutical ingredients or "APIs") and commissioned oral bioavailability studies for these drug substances. For example, I worked on the design of suitable formulations of these novel drug substances, experimental protocols for bioavailability studies including selection of excipients, and interpretation of the results of bioavailability studies.

22. Evidence of the impact of my publications comes from the following:

- My publications have been cited approximately 65,000 times, including more than 18,800 citations since 2020;
- My h-index is 120 which means that 120 of my publications have been cited at least 120 times;
- On February 10, 2011, I was listed by Thomson Reuters as the 20th most impactful chemist in the world for work published between 2000 and 2010; and

- In 2014, 2015, 2016, 2018, 2019, and 2022 I was listed as a "highly cited researcher" by Thomson Reuters (and then Clarivate Analytics) for the number of highly cited articles published in the previous decade. Worldwide, approximately 200 chemists receive this honor each year.

23. I have delivered more than 280 invited presentations at national and international meetings in the area of crystal structures and crystal engineering, including frequent keynote or plenary lectures at international meetings. Approximately 100 of these presentations addressed aspects of the crystal structures of drug substances with particular emphasis upon understanding crystal packing and physicochemical properties in different types of their crystal forms such as polymorphs, salts, hydrates, solvates, and cocrystals.

24. I have presented approximately 220 invited seminars at companies, universities, and government laboratories in North America, Asia, Africa, Europe, and South America, most of which addressed fundamental and applied aspects of crystal engineering.

25. I have consulted for pharmaceutical companies in the United States, Europe, and India. I have also presented lectures specifically on the subjects of polymorphism and solid forms of drug substances at industrial workshops and conferences. From 2003 to 2005, I served on the Scientific Advisory Board of a company that developed novel forms and formulations of drug substances, which Johnson and Johnson (Transform Pharmaceuticals) purchased. I have also served as Scientific Advisor to a company that develops novel forms and formulations of drug substances (Thar Pharmaceuticals) and to a company that develops new drug products (Alkermes). During this consulting work, I have advised companies about novel drug substances such as pharmaceutical cocrystals or salts and how to formulate them as potential drug products as part of pre-clinical and clinical drug development research programs. I have also advised companies about FDA guidelines in the context of solid drug substances and their formulations. I have also

conducted testing of drug substances and drug products in terms of their identity, stability, and water content.

26. My current research program focuses on crystal engineering, the field of chemistry that studies the design, properties, and applications of crystalline materials. At the time I started my independent career in 1985, I was one of just a handful of researchers in the field. Research then addressed basic design principles in molecular and network solids. Today, the focus of crystal engineering has shifted to properties and applications.

27. My credentials and additional details pertaining to the above are fully set out in my curriculum vitae which is attached hereto as Appendix 2.

III. DOCUMENTS AND MATERIALS REVIEWED

28. Appendix 1 at the end of this declaration lists the materials I considered in preparing this declaration. I also relied on my personal knowledge and over 42 years of professional experience in subject matter of relevance to the Polymorph Patents.

IV. PERSON OF ORDINARY SKILL IN THE ART ("POSA")

29. I understand that a POSA is a hypothetical person who is presumed to have knowledge of all of the relevant art at the time of the alleged invention. I am informed that factors that may be considered in determining the level of ordinary skill in the art may include: (1) type of problems encountered in the art; (2) prior art solutions to those problems; (3) how quickly innovations are made; (4) sophistication of the technology; and (5) educational level of active workers in the field. In a given case, not every factor may be present, and one or more factors may predominate. I am further informed that the qualifications of the POSA may be met by an individual or by a team of individuals. I am also informed that a POSA is a person of ordinary creativity, and would have the capability of understanding the scientific principles applicable to the pertinent art.

30. I understand that Defendants have previously provided the following definition of a POSA in their Invalidity Contentions: a POSA would have experience with the synthesis and manufacture of active pharmaceutical ingredients and the characterization of their physicochemical properties. A POSA would include, for example, an individual with a B.S. or M.S. degree in pharmaceutical sciences, chemistry, chemical engineering, solid-state physics, or similar discipline related to the solid state chemistry of drug substances. The POSA would also most likely have at least 3-5 years of experience in the pharmaceutical industry and would have a working knowledge of the evaluation and modification of the physicochemical properties of drug substances and the formulation of drug substances. The POSA may also have a Ph.D. in one of the disciplines named above or a similar discipline. Greater work experience may substitute for a more advanced degree.

31. Defendants' definition of a POSA is consistent with my understanding of the definition of a POSA for the Polymorph Patents based on my review of the Polymorph Patents, experience working in the crystal solids industry and engineering, supervising students, publishing, and conducting all of my research over four decades. I further clarify that an individual with a B.S. or M.S. degree would necessarily need more years of work experience to qualify as a POSA, in comparison to an individual with a Ph.D. I also clarify that a POSA would further have access to individuals with pertinent knowledge and skill he or she did not possess, including a medical doctor.

32. I understand Plaintiffs have previously provided the following definition of a POSA: A person with an advanced degree, such as a M.S., Ph.D., or equivalent, in chemistry, chemical engineering, or a related field, with at least two years of laboratory experience working with pharmaceutical solids, including polymorphic forms; or alternatively, a Bachelor's degree in

chemistry, chemical engineering, or a related field, with at least five years of practical experience working with pharmaceutical solids, including polymorphic forms.

33. I consider myself to be a POSA under either definition based on my qualifications and experience described above and in my curriculum vitae with respect to the subject matter in the Polymorph Patents as of at least 2009.

V. LEGAL STANDARD FOR CLAIM CONSTRUCTION

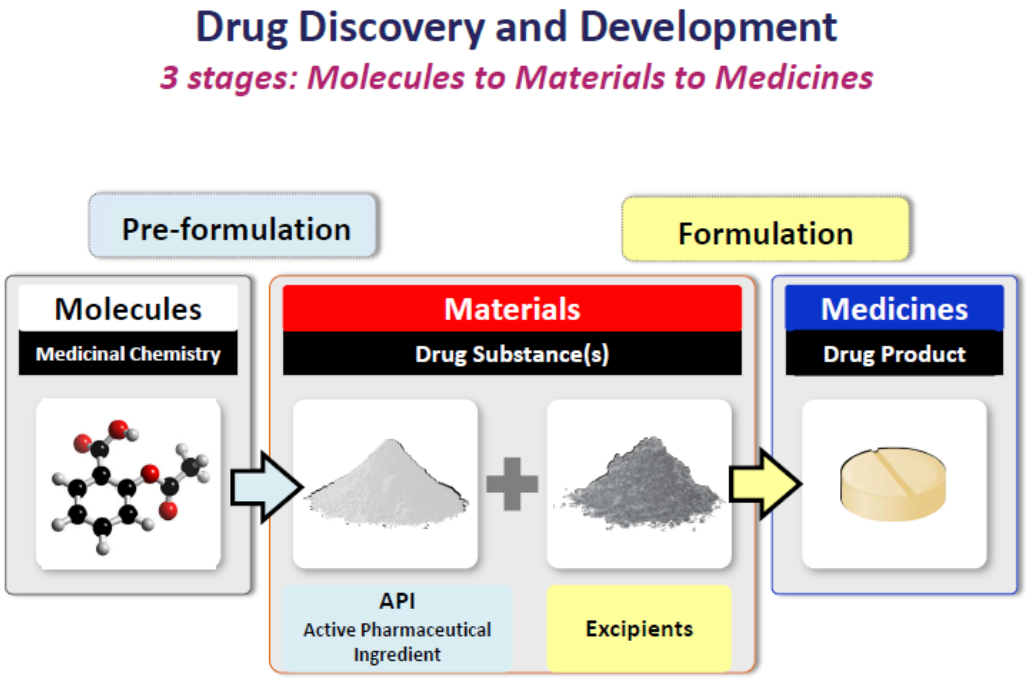
34. I have been informed by Defendants' counsel that patent claim terms are construed from the perspective of a POSA as of the effective filing date of the patent application. I understand from Defendants' counsel that the earliest possible priority date of the Polymorph Patents is July 15, 2009, and that I should utilize this date for the purposes of my analysis.

35. I have been further informed by Defendants' counsel that claim terms should be considered in the context of the entire patent claim in which they appear, as well as in the context of the other claims, the specification, and the prosecution history of the patent, as opposed to in isolation or out of context.

36. I have also been informed by Defendants' counsel that it may be appropriate to consider evidence outside of the patent and its prosecution history, provided this extrinsic evidence is not used to alter or contradict the meaning established by the intrinsic evidence of the patent and its prosecution history.

VI. TECHNICAL BACKGROUND

37. Generally speaking, drug development can be divided into three main steps or subsections: the "molecules" step; the "materials" step; and the "medicines" step. The diagram below, which I prepared, illustrates these three general steps.



38. The "molecules" step involves drug discovery where medicinal chemists prepare potential drug molecules and determine which ones are efficacious against the intended biological target.

39. The "materials" step comes after a drug molecule has been chosen as a candidate for possible use as the active drug substance in a drug product and includes identifying a solid form or forms of the selected drug compound (e.g., a salt of the drug molecule, a cocrystal of the drug molecule or, if the molecule is basic, the freebase itself) suitable for use in a drug product. These solid forms then become candidate drug substances that are characterized by appropriate testing methods that focus upon identification and determination of their physicochemical properties. Once suitable solid forms of the drug compound are identified through routine screening experiments and analytical testing, they are considered for use as drug substances (active pharmaceutical ingredients, APIs).

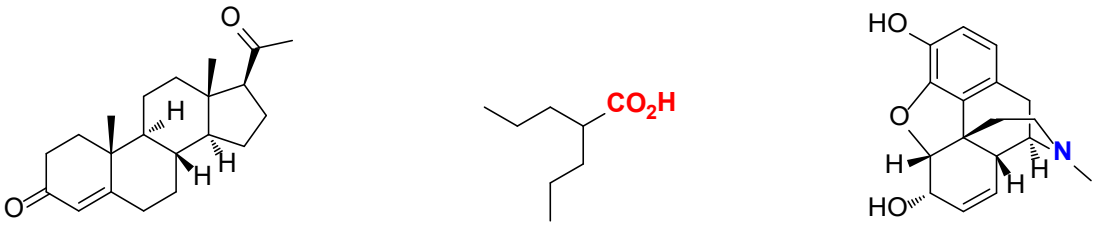
40. The "medicines" step includes the formulation stage of drug development and involves analysis of the types of dosage forms, such as tablets, capsules, etc., that can be used to

formulate the selected drug substance with inactive excipients that enable the selected drug substance to be used in a drug product.

A. SOLID FORMS OF DRUG COMPOUNDS

41. Once a drug molecule is selected for further study at the "molecules" stage, it is necessary to first identify a suitable drug compound, and then, a suitable solid form of the drug compound to serve as a drug substance. Solid form selection includes a decision as to whether to use a crystalline or amorphous form, as well as other considerations, such as whether particle size will impact dissolution. The drug molecule might be inherently non-ionizable, or it might possess a suitably ionizable acidic or basic group, in which case it may then be referred to as the "free acid" or "free base", respectively. This type of drug compound is exemplified below with one example of each type of drug molecule: progesterone, a non-ionizable molecule (*i.e.*, the drug molecule does not have acid or base functionality); valproic acid, a free acid (colored red); and morphine, a free base (colored blue).[1]

Progesterone (non-ionizable) **Valproic acid (free acid)** **Morphine (free base)**



42. After identifying and selecting a suitable drug compound, the pharmaceutical scientist would conduct further routine screening studies to identify a suitable solid (most likely, crystalline) form of the selected drug compound.

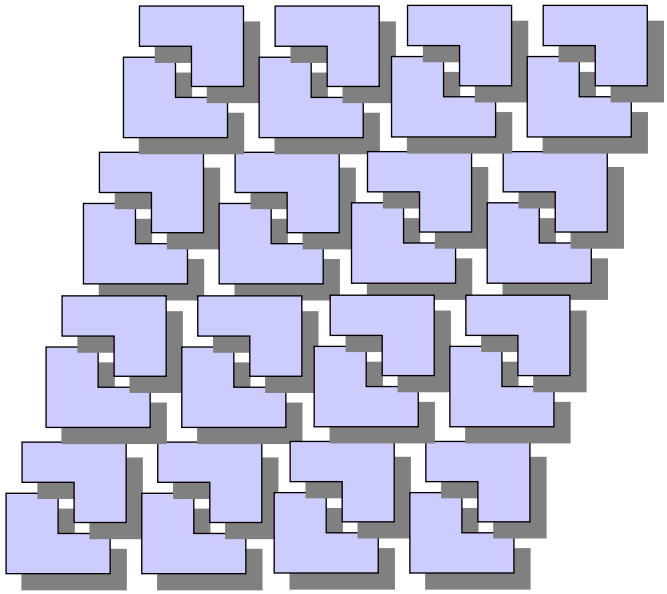
[1] The acidic group (**CO_2H**) in valproic acid and the basic tertiary amine group (**N**) in morphine have been highlighted in red and blue, respectively. In other basic drugs, the ionizable amine group may be a primary (NH_2) or secondary (NH) group.

43. A crystalline solid is a solid in which the individual molecules, atoms, or ions are arranged in a repeating pattern which extends over a long range in three dimensions. This is described in many industry references, including, for example, Ex. AB (Myerson & Ginde 2002) at DEFSREV0007876, Ex. Z (Byrn 1999) at DEFSREV0000700, Ex. Y (Cullity & Stock 2001) at DEFSREV0007766, and Ex. AA (Ohannesian 2002) at DEFSREV0008162. A characteristic property of a crystal is that it has a well-defined melting point; the temperature at which the crystal lattice breaks down and the crystalline solid becomes liquid. A crystal's melting point is a crystal characteristic routinely described in pharmaceutical science publications, such as, for instance, Ex. AD (Haleblian 1969) at DEFSREV0001515.

44. Crystalline solids are also characterized by exhibition of sharp diffraction peaks at defined positions. A testing method known as X-ray Powder Diffraction or "XRPD" is routinely used to determine whether or not a solid is a crystalline solid as discussed in detail below. The testing method is widely used and described in the art, such as, for example Ex. AC (Brittain 1999) at DEFSREV0000599-DEFSREV0000601.

45. In crystalline solids the molecules, atoms, or ions interact with each other to form chemical bonds of different kinds such as noncovalent bonds (molecular solids), ionic bonds (ionic solids), or covalent bonds (network solids). This is taught to undergraduate students and explained in, for instance, Ex. Z (Byrn 1999) at DEFSREV0000702. Examples of commonly recognized crystalline solids are ice, diamond, and sodium chloride, in which the interactions are noncovalent bonds, covalent bonds and ionic bonds, respectively. Almost all drug compounds are classified as organic compounds and therefore exist as molecular solids, as further explained by Ex. Z (Byrn 1999) at DEFSREV0000698-DEFSREV0000739.

46. Crystalline solids tend to exhibit a high degree of purity, and they can typically be processed at an industrial scale. That crystalline solids form ordered arrangements that repeat in three dimensions means that they can be depicted as shown below in schematic fashion in a diagram that I prepared.

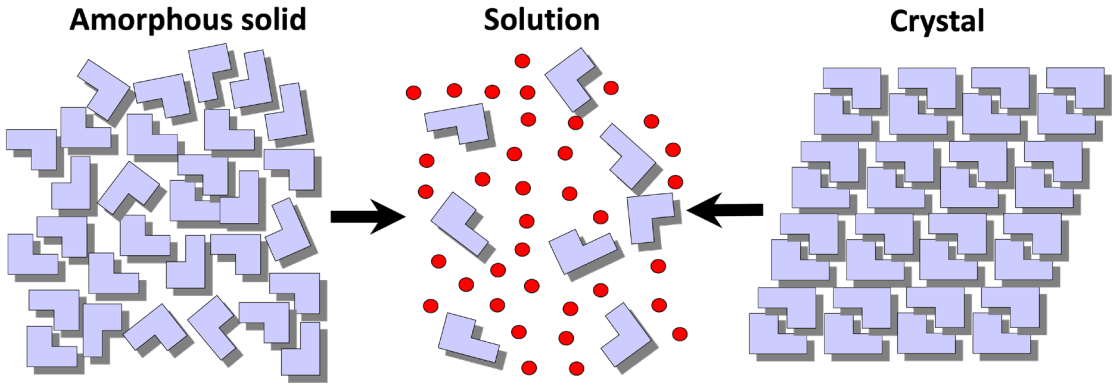


B. CRYSTALLIZATION

47. Crystallization from solution (solvent-mediated crystallization) is known as a method that, among other things, is reproducible and increases the consistency with which a drug compound can be purified and processed. In addition, crystalline forms of drug compounds are generally regarded to be most desirable due to their physical and chemical stability when compared to the corresponding amorphous form in which there is no repeating pattern of molecules, ions or atoms over long range. *See*, *e.g.,* Ex. AC (Brittain 1999) at DEFSREV0000596 ("*amorphous* [solids are] solid forms that have no long-range molecular order"). Therefore, all else being equal, researchers in the pharmaceutical industry have long preferred to work with crystalline solids and routinely crystallized the drug compounds with which they worked.

48. The general technique of crystallization is taught (and has long been taught) to undergraduate students as part of their training and is used routinely by chemists in the preparation and purification of organic compounds. The general procedure to crystallize chemical compounds has been described in numerous standard texts on laboratory methods, such as Fessenden (*See* Ex. X (Fessenden 1983), among many others.

49. The process of solvent-mediated crystallization starts with dissolution of a solid in a liquid to create a solution as illustrated in the following schematic diagram which I prepared.



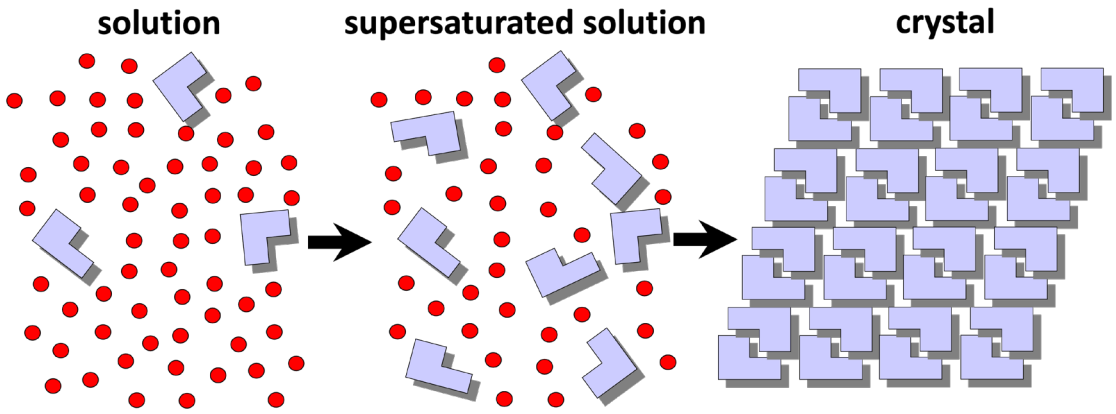
50. When a solid is mixed with a liquid and dissolved it becomes part of this liquid if the amount of solid is below the solubility of that solid in the liquid. After dissolution, the solid is then called the solute and the liquid is called the solvent. If the amount of solid is above the solubility of that solid in the liquid, then heating and/or addition of more liquid can be conducted to form the solution. Since a solution has no long range order (the solute molecules or ions are randomly dispersed), there is no memory of the solid structure of the solute that was present before it was dissolved.

51. Solvent-mediated crystallization is typically induced by changing the state of the system[2] in some way that reduces the solubility of the crystallizing compound in a controlled

[2] The "system" is the drug compound dissolved in at least one solvent. Other compounds and solvents may or may not be present in the system.

manner. This change causes the system to become supersaturated — there is more solute[3] in the system than is soluble in the solvent. Supersaturation is most commonly induced by slowly evaporating the solvent or cooling the solution in a controlled fashion. Supersaturation is also commonly induced by, for example, solvent diffusion, vapor diffusion, changing the polarity of the solvent (such as by adding an anti-solvent), changing the pH of the solution (by adding acid or base), or salting out (a type of common ion effect). Ex. X (Fessenden 1983) at DEFSREV0008079.

52. The use of supersaturation to induce crystallization is illustrated in the following schematic diagram which I prepared:



53. If a crystallization process is not conducted under conditions that control the rate of supersaturation and prevent the solution from becoming supersaturated too quickly, an amorphous solid may result or the crystallization process may not be reproducible. For this reason, slow cooling and slow evaporation were, and are, the two primary and first-tried methods of crystallization used in a crystal form screening experiment. Both of these processes can be controlled to occur gradually, which is highly desirable in any procedure calculated to lead to a

[3] The "solute" in this case is the drug compound.

thermodynamically stable crystal form.[4] Conversely, more rapid cooling and evaporation would be expected to lead to less stable crystal forms (also referred to as kinetically stable or metastable crystal forms) or, if very rapid, an amorphous form.

54. Because solubility is a key parameter to many crystallization methods, the first step in a routine crystallization experiment is to systematically determine a proper solvent system. The solute must be soluble enough to allow complete dissolution in the solvent system but not so soluble as to prevent or inhibit crystallization.

55. When attempting to crystallize a compound for the first time, a person of ordinary skill would have used different approaches to determine the conditions that are most favorable to crystallization. This exercise would have been taught to persons of ordinary skill in the art as part of their education and training. Thus, one of ordinary skill in the art would try, for example, several different solvents at different rates of evaporation and cooling. After observing how the material behaves, a person of ordinary skill in the art would further refine these experiments to optimize the crystallization. Finally, one would use routine analytical techniques such as XRPD, DSC, or TGA, which are described below, to characterize the products of a crystallization screen. Serially conducting numerous such experiments to see how the substance behaves under different conditions was and is routine in the field of crystallization. In fact, it is through such a routine

[4] A thermodynamically stable crystalline form is the most stable crystalline form under a given set of conditions, that is, it will not spontaneously convert to another form under those specific conditions. In general, it is preferred to use a solid form of a drug compound that is thermodynamically stable under ambient conditions of temperature and pressure when developing a drug product since this eliminates the risk of having a metastable form convert to a more stable form during manufacturing or storage, which has the potential to affect the performance of a drug product. A drug compound can have more than one thermodynamically stable crystalline form if conditions change, for example, a hydrated solid form might be stable under humid conditions, and an anhydrous solid form under dry conditions.

approach that different crystalline solids (i.e., polymorphs, solvates, hydrates and the like) can be made and characterized.

56. Generally, all small molecules that are solid at ambient conditions form at least one crystalline solid. This is especially true for molecules with features that promote hydrogen bonding, i.e., the vast majority of drug compounds, and thus facilitate crystal packing. By the mid-1980s, a chemist, or a similarly skilled individual, would reasonably have expected that the procedures described above would successfully result in the preparation of crystalline solids for any small molecule that was known to be crystalline under ambient conditions. This would be particularly true for those compounds that have a propensity to form intermolecular hydrogen bonds, which contain numerous hydrogen bond donors and hydrogen bond acceptors.

57. After an appropriate solvent system for the crystallization has been selected, subsequent recrystallizations can utilize crystal seeding and/or Ostwald ripening to produce a consistent crystalline form of a drug compound. Seeding involves the induction of crystallization from a solution by providing a starting spot (the "seed") for the crystal lattice. This seed provides the molecules in the solution a point around which to propagate the crystalline lattice of a specific crystalline form.[5] Ostwald ripening involves prolonged stirring during the crystallization process. During this extended time, smaller crystals dissolve, and the dissolved molecules then recrystallize on the surface of larger crystals in the same manner as a seeded solution. This approach, also called slurrying, can allow for the preparation of a more uniform crystalline solid, which is desirable in large-scale preparations, such as for manufacture of drug substances.

[5] If the seed of a solvate (or hydrate) is utilized, there must be sufficient solvent (or water) present in solution for seeding to produce more crystals of the solvate (or hydrate).

C. IDENTIFICATION AND CHARACTERIZATION OF CRYSTALLINE SOLIDS

58. A crystalline solid can be identified by its inherent characteristics such as its crystal structure, which refers to the identity, number, and position of the atoms relative to each other extending in three dimensions, along with the packing arrangement and symmetry elements of the structure that define the repeating units. A number of techniques were known in the art as of 2009 to characterize crystalline solids, some of which are discussed below. Each of these methods are included in standard references, such as Remington's Pharmaceutical Sciences and the United States Pharmacopeia.

59. The United State Pharmacopeia ("USP") is a compendium of information about drug substances and drug products that is published annually as a combined volume with the National Formulary ("NF"). The resulting USP-NF is an authoritative document used by pharmaceutical scientists as it sets written standards for medicines and their ingredients, including how to conduct physical tests that provide information about their purity and identity. The USP provides multiple sections that discuss characterization of drug substances: Section 941 ("X-Ray Diffraction"); Section 891 ("Thermal Analysis"). Ex. AE (USP 2008) at DEFSREV0007974-DEFSREV0007976, DEFSREV0007972, DEFSREV0007973.

60. Remington's Pharmaceutical Sciences is a textbook and reference guide that is used in pharmaceutical sciences. Remington's explains that differential scanning calorimetry and powder x-ray diffraction are common tools used to characterize polymorphs. Ex. U (Remington's 2000) at DEFSREV0001884.

1. X-Ray Powder Diffraction

61. The ability of crystalline solids to diffract X-rays has been known since 1912 and makes possible a number of experimental testing methods that can identify crystalline solids and

determine attributes of a crystal structure. Powder X-ray diffraction (or X-ray powder diffraction), PXRD (or XRPD), can be used to identify bulk crystalline and microcrystalline solids and is typically conducted on a powder sample that contains randomly oriented microcrystals. XRPD is generally regarded as the "gold standard" for identification of the crystal form of a drug compound since it can produce a pattern of peaks that acts as a signature, or fingerprint, for a particular crystalline solid. The theory and operation of XRPD has been well-known in the art for decades. *See, e.g.*, Ex. AD (Haleblian 1969) at DEFSREV0001522; Ex. AC (Brittain 1999) at DEFSREV0000599-DEFSREV0000601. XRPD's relevance to identification of the crystalline form of drug compounds is therefore long and well-established.

62. In XRPD, X-rays are directed at a powder sample at varying angles. If the sample is crystalline, it will diffract X-rays at specific angles because of its regular, repeating arrangement of molecules or ions. An XRPD pattern typically displays as an X-Y plot of intensity (measured in counts) vs. diffraction angle (measured in units of degrees 2 theta (2θ)). A given crystalline solid will exhibit a characteristic pattern of peaks like a fingerprint, generally represented by as many peaks as can be identified. Peak positions are typically quoted by their 2-theta value or the d-spacing value that corresponds to this 2-theta value.

63. Whereas an XRPD pattern is an inherent characteristic of a solid form, as discussed above, XRPD is unsuitable for determining if a crystalline solid contains water molecules (hydrate), solvent molecules (solvate) or not (anhydrate) and different crystal forms of the same chemical compound can exhibit similar XRPD patterns. XRPD is therefore not definitive about the identity of a crystal form and is typically used in combination with other analytical tests. Nevertheless, whereas a XRPD pattern revealing sharp peaks requires secondary testing to verify its identity, XRPD revealing diffuse peaks (an amorphous halo) is considered definitive that a solid

has no long range order and is amorphous. In other words, while other analytical tests may provide additional information concerning an amorphous solid—e.g., water associated with the pharmaceutical compound, such as loose surface water—such testing does not overcome the clear results of XRPD. If no sharp and well-defined XRPD peaks are present, the tested powder is not classified as a crystalline solid.

2. Melting Point and Differential Scanning Calorimetry

64. Differential scanning calorimetry ("DSC") measures exothermic (heat is released) and endothermic (heat is absorbed or added to the system) thermal events as a substance is slowly heated. Crystalline solids, for instance, exhibit an endothermic peak over a relatively short temperature range at their melting point.

65. Other types of thermal event related to phase changes in crystalline solids can be observed from DSC. For example, loss of water or solvent molecules tends to result in an endothermic peak over a relatively broad temperature range whereas transformation to a more stable crystal solid or a chemical reaction will afford an exothermic peak.

66. DSC "can also be used to establish the melting points of polymorphic species." Ex. AC (Brittain 1999) at DEFSREV0000608. The melting point is a characteristic property of a solid substance.

3. Thermogravimetric Analysis

67. Thermogravimetric analysis ("TGA") measures the weight change of a substance as it is slowly heated. TGA provides a quantitative measure of the amount and rate of change in the weight of a substance as a function of temperature or as a function of time in a controlled atmosphere. TGA "is a useful method for the quantitative determination of the total volatile content of a slid and can be used as an adjunct to Karl Fischer titrations for the determination of moisture." Ex. AC (Brittain 1999) at DEFSREV0000604. It is used primarily to provide

information about the composition of materials and to determine their thermal stability at temperatures up to 1,000ºC. TGA can be useful for determining if water molecules (hydrate) or solvent molecules (solvate) are present in a solid compound and how much water or other solvent molecules are present in a sample. When TGA is coupled with a technique such as mass spectroscopy (TGA/MS) it is possible to determine which specific solvent(s) might be present in a solid form.

D. PHARMACEUTICAL FORMULATIONS

68. Turning to the "medicines" stage, the next stage of drug development includes determining the types of dosage forms suitable for a given drug compound. Typical dosage forms used that comprise crystalline solids include tablets and capsules, where the crystalline form remains intact. Other dosage forms may be used, such as solutions, but it is understood that once dissolved, the solution will have no memory of the source of the crystalline solid.

69. The USP provides an overview of dosage forms. With respect to solutions, the USP explains that "[s]olutions are liquid preparations that contain one or more chemical substances dissolved, i.e. molecularly dispersed, in a suitable solvent or mixture of mutually miscible solvents." Ex. AE (USP 2008) at DEFSREV0007983. The USP further explains "[s]ince molecules in solutions are uniformly dispersed, the use of solutions as dosage forms generally provides for the assurance of uniform dosage upon administration and good accuracy when diluting or otherwise mixing solutions." Ex. AE (USP 2008) at DEFSREV0007983. Remington similarly explains that "[a] solution, in the present context, is a liquid preparation that contains one or more soluble chemical substances dissolved in water." Ex. U (Remington 2000) at DEFSREV0007916. In a solution dosage form, there is no memory of the solid form of the drug compound used to prepare that solution.

70. In contrast, the USP defines suspensions as "liquid preparations that consist of solid particles dispersed throughout a liquid phase in which the particles are not soluble." USP 2008 at 623. Remington, relying on the USP, also explains that the definitions of suspensions are "formulated in such a way that an insoluble substance is suspended in a liquid at some stage of the manufacturing or dispersing process." Ex. U (Remington 2000) at DEFSREV0007940. In suspensions, the crystalline solid would remain intact.

VII. THE POLYMORPH PATENTS AND THEIR PROSECUTION HISTORIES

A. THE '451 PATENT

71. The '451 patent has the title "Crystalline freebase forms of a biphenyl compound," and issued on September 24, 2013. The '451 patent lists Grahame Woollam as the inventor and Theravance Inc. as the assignee. *See* Ex. A, '451 patent at Cover. The '451 patent issued from U.S. Application No. 12/835,964 ("the '964 application"), which was filed on July 14, 2010.

72. The '451 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

73. I understand that the asserted claims of the '451 Patent are 1–8, and 12, of which claims 1 and 5 are independent and directed to Forms III and IV of revefenacin,[6] respectively. The claims are as follows:

> 1. A crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1; designated as Form III; and having a melting point of about 125° C.

[6] 1-(2-{4 [(4-carbamoylpiperidin-1-yl)methyl]-N-methylbenzamido}ethyl)piperidin-4-yl N-({1,1' biphenyl}-2-yl)carbamate is the chemical name for revefenacin.

2. The crystalline compound of claim 1, characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values selected from 6.6±0.1, 11.4±0.1, 13.1±0.1, 16.1±0.1, 17.5±0.1, 18.2±0.1, 18.6±0.1, 19.3±0.1, 19.7±0.1, 19.9±0.1, 20.2±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.

3. The crystalline compound of claim 1, further characterized by a powder x-ray diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in FIG. 1.

4. The compound of claim 1, further characterized by a differential scanning calorimetry thermogram substantially in accordance with that shown in FIG. 4.

5. A crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 10.6±0.1, 15.0±0.1, 16.0±0.1, 17.3±0.1, 17.7±0.1, 20.9±0.1, 21.4±0.1, 22.6±0.1, 24.6±0.1, and 27.8±0.1; designated as Form IV; and having a melting point of about 119° C.

6. The crystalline compound of claim 5, further characterized by a powder x-ray diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in FIG. 2.

7. The compound of claim 5, further characterized by a differential scanning calorimetry thermogram substantially in accordance with that shown in FIG. 5.

8. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and the compound of claim 1.

12. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and the compound of claim 5.

74. The ’964 application was filed on July 14, 2010. On November 29, 2012, the examiner rejected all claims over prior art. Ex. B, ’451 patent prosecution history, Nov. 29, 2012 Non-Final Rejection PLTF-YUP-00000533-PLTF-YUP-00000554. The applicant responded on February 27, 2013; the applicant amended claim 1 to recite that the crystalline freebase was selected from Form III having a melting point of about 125° C and Form IV having a melting point of about 119° C. Ex. B, ’451 patent prosecution history, Feb. 27, 2013 Response to Non-Final

Rejection at PLTF-YUP-00000654-PLTF-YUP-00000657. The applicant also argued that Forms I and II of Axt are produced by the examples of Mammen '133 and exhibit different X-ray powder diffraction (XRPD) peaks and lower melting points than the claimed Forms III and IV. Ex. B, '451 patent prosecution history, Feb. 27, 2013 Response to Non-Final Rejection PLTF-YUP-00000654-PLTF-YUP-00000657. The examiner allowed the claims, with an amendment, and explained "there was no prior art which teaches or suggests [revefenacin] in crystalline freebase form III or form IV, with a characteristic x-ray diffraction peaks as cited in the claims," having certain melting points. Ex. B, '451 patent prosecution history, May 28, 2013 Notice of Allowance at PLTF-YUP-00000675.

B. THE '028 PATENT

75. The '028 patent has the title "Crystalline freebase forms of a biphenyl compound," and issued on September 19, 2017. The named inventor is Grahame Woollam and the assignee is Theravance Biopharma R&D IP, LLC. Ex. G, '028 patent at Cover. The '028 patent issued from U.S. Application No. 15/206,877 ("the '877 application"), which was filed on July 11, 2016. The '028 patent is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed December 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed November 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8.921,396), filed August 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010.

76. I understand that the '028 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

77. The '028 patent contains six independent claims, all of which I understand are currently asserted against Defendants. The claims read as follows:

> 1. Crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and
>
> further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
>
> 2. Crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values selected from 6.6±0.1, 11.4±0.1, 13.1±0.1, 16.1±0.1, 17.5±0.1, 18.2±0.1, 18.6±0.1, 19.3±0.1, 19.7±0.1, 19.9±0.1, 20.2±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
>
> 3. A pharmaceutical composition comprising:
>
> (a) a pharmaceutically acceptable carrier; and(b) crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]-methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
>
> 4. A pharmaceutical composition comprising:
>
> (a) a pharmaceutically acceptable carrier; and
>
> (b) crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]-methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values selected from 6.6±0.1, 11.4±0.1, 13.1±0.1, 16.1±0.1, 17.5±0.1, 18.2±0.1, 18.6±0.1, 19.3±0.1, 19.7±0.1, 19.9±0.1, 20.2±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
>
> 5. Crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern having peak positions in accordance with the peak positions shown in FIG. 1.
>
> 6. A pharmaceutical composition comprising:

(a) a pharmaceutically acceptable carrier; and

(b) crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyfl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern having peak positions in accordance with the peak positions shown in FIG. 1.

78. The ’877 application was filed on July 11, 2016. The claims were rejected in a non-final rejection, and the applicant responded. The examiner allowed the claims and explained “there is no prior art which teaches or suggests the freebase crystalline form III of [revefenacin], with the characteristic x-ray diffraction peaks as cited in the claims.” Ex. H, ’028 Patent prosecution history, May 25, 2017 Notice of Allowance at PLTF-YUP-00001852.

C. THE ’013 PATENT

79. The ’013 patent, entitled “Crystalline freebase forms of a biphenyl compound,” issued on October 16, 2018, and is assigned on its face to Theravance Biopharma R&D IP, LLC. The named inventor is Grahame Woollam. Ex. O, ’013 patent at Cover. The ’013 patent issued from U.S. Application No. 15/677,264 (“the ’264 application”), which was filed on August 15, 2017. The ’013 patent is a continuation of U.S. Application No. 15/206,877 (issued as the ’028 patent), filed July 11, 2016, which is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed December 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed November 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8,921,396), filed August 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010. The ’028 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

80. I understand that the asserted claims of the ’013 patent are claims 1–9, where claim 1 is independent. The claims are as follows:

1. A method for preparing a pharmaceutical composition for use in a nebulizer inhaler, the method comprising dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in an aqueous pharmaceutical carrier to form an aqueous solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

2. The method of claim 1, wherein the crystalline freebase is further characterized by five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.

3. The method of claim 1, wherein the crystalline freebase is further characterized by a powder x-ray diffraction pattern having peak positions in accordance with the peak positions shown in FIG. 1.

4. The method of claim 1, wherein the crystalline freebase is further characterized by a melting point of about 125° C.

5. The method of claim 1, wherein the crystalline freebase is further characterized by a differential scanning calorimetry thermogram in accordance with that shown in FIG. 4.

6. The method of any one of claims 1 to 5, wherein the aqueous solution is isotonic.

7. The method of any one of claims 1 to 5, wherein the aqueous solution has a pH of about 4-6.

8. The method of any one of claims 1 to 5, wherein the aqueous solution is buffered with citrate buffer to a pH of about 5.

9. The method of any one of claims 1 to 5, wherein the aqueous solution contains about 0.05 μg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester.

81. The ’264 application was filed on August 15, 2017. The claims were rejected in a non-final rejection, and the applicant responded. The examiner allowed the claims and explained “the prior art does not teach or suggest a method of preparing an aqueous solution comprising crystalline freebase of [revefenacin] characterized by a powder x-ray diffraction pattern comprising” specific diffraction peaks. Ex. P, ’013 patent prosecution history, June 15, 2018 Notice of Allowance at PLTF-YUP-00002218-PLTF-YUP-00002219.

D. THE '081 PATENT

82. The '081 patent, entitled "Crystalline freebase forms of a biphenyl compound," issued on February 4, 2020, and is assigned on its face to Theravance Biopharma R&D IP, LLC. The named inventor is Grahame Woollam. Ex. M, '081 patent at Cover. The '081 patent issued from U.S. Application No. 16/130,079 ("the '079 application"), which was filed on September 13, 2018. The '081 patent is a continuation of U.S. Application No. 15/677,264 (issued as the '013 patent), filed August 15, 2017, which is a continuation of U.S. Application No. 15/206,877 (issued as the '028 patent), filed July 11, 2016, which is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed Dec. 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed Nov. 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8,921,396), filed Aug. 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010. The '028 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

83. The '081 patent has four independent claims, all of which I understand are currently asserted against the Defendants. The claims of the '081 patent read as follows:

> 1. A crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester having a melting point of about 125° C.
>
> 2. A crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester having a differential scanning calorimetry thermogram in accordance with that shown in FIG. 4.
>
> 3. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester having a melting point of about 125° C.

> 4. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester having a differential scanning calorimetry thermogram in accordance with that shown in FIG. 4.

84. The '079 application was filed on September 13, 2018. The claims were rejected in a non-final rejection, and the applicant responded. In response, the applicant argued the prior art "did not find the presently claimed crystalline freebase form having a highly desirable, significantly higher melting point" and that "[t]he presently claimed crystalline freebase form is prepared under different conditions than those used [in the prior art] and it has different physical properties, i.e., a significantly higher melting point." Ex. N,'081 patent prosecution history, Jul. 10, 2019 Response to Office Action at PLTF-YUP-00002543, PLTF-YUP-00002545. The examiner allowed the claims and explained "the claimed crystalline freebase of [revefenacin] having a melting point of about 125 °C, and the differential scanning calorimetry thermograph in accordance with that shown in Fig. 4, corresponding to polymorph form III is not taught or suggested by the closest prior art." Ex. N, '081 patent prosecution history, Sept. 17, 2019 Notice of Allowance at PLTF-YUP-00002560-PLTF-YUP-00002570.

E. THE '289 PATENT

85. The '289 patent, entitled "Crystalline freebase forms of a biphenyl compound," issued on May 18, 2021, and is assigned on its face to Theravance Biopharma R&D IP, LLC. The named inventor is Grahame Woollam. Ex. I, '289 patent at Cover. The '289 patent was filed on December 16, 2019 as U.S. Application No. 16,715,225 ("the '225 application"). The '289 patent is a continuation of U.S. Application No. 16/130,079 (issued as the '081 patent), filed, Sept. 13, 2018, which is a continuation of U.S. Application No. 15/677,264 (issued as the '013 patent), filed on August 15, 2017, which is a continuation of U.S. Application No. 15/206,877 (issued as the

'028 patent), filed July 11, 2016, which is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed Dec. 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed Nov. 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8,921,396), filed Aug. 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010. The '028 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

86. The '289 patent has nine claims, all of which I understand are currently asserted against Defendants, and of which claim 1 is the only independent claim. The claims of the '289 patent read as follows:

> 1. A method for treating chronic obstructive pulmonary disease in a human patient, the method comprising:
>
> (a) preparing a pharmaceutical composition by dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in an aqueous pharmaceutical carrier; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and
>
> (b) administering the pharmaceutical composition to the human patient using a nebulizer.
>
> 2. The method of claim 1, wherein the crystalline freebase is further characterized by five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
>
> 3. The method of claim 1, wherein the crystalline freebase is further characterized by a powder x-ray diffraction pattern having peak positions in accordance with the peak positions shown in FIG. 1.
>
> 4. The method of claim 1, wherein the crystalline freebase is further characterized by a melting point of about 125° C.

> 5. The method of claim 1, wherein the crystalline freebase is further characterized by a differential scanning calorimetry thermogram in accordance with that shown in FIG. 4.
>
> 6. The method of any one of claims 1 to 5, wherein the pharmaceutical composition is isotonic.
>
> 7. The method of any one of claims 1 to 5, wherein the pharmaceutical composition has a pH of about 4-6.
>
> 8. The method of any one of claims 1 to 5, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.
>
> 9. The method of any one of claims 1 to 5, wherein the pharmaceutical composition contains about 0.05 µg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1424 [4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester.

87. The ’225 application was filed on December 16, 2019. The claims were rejected in a non-final rejection, and the applicant responded. The examiner allowed the claims and explained “crystalline freebase form of [revefenacin] having a powder X-ray diffraction pattern comprising” specific diffraction peaks, in accordance with that shown in Fig. 1 (form III as defined in Applicant’s specification) is not taught or suggested by the closest prior art. Ex. J, ’289 patent prosecution history, Jan. 25, 2021 Notice of Allowance at PLTF-YUP-00002877- PLTF-YUP-00002878.

F. THE ’209 PATENT

88. The ’209 patent, entitled “Crystalline freebase forms of a biphenyl compound,” issued on May 16, 2023, and is assigned on its face to Theravance Biopharma R&D IP, LLC. The named inventor is Grahame Woollam. Ex. K, ’209 patent at Cover. The ’209 patent was filed on June 15, 2022 as U.S. Application No. 17/841,567 (“the ’567 application”). The ’209 patent is a continuation of U.S. Application No. 17/301,820 (issued as the ’948 patent), filed April 15, 2021, which is a continuation of U.S. Application No. 16/715,255 (issued as the ’289 patent), filed December 16, 2019, which is a continuation of U.S. Application No. 16/130,079 (issued as the

'081 patent), filed September 13, 2018, which is a continuation of U.S. Application No. 15/677,264 (issued as the '013 patent), filed on August 15, 2017, which is a continuation of U.S. Application No. 15/206,877 (issued as the '028 patent), filed July 11, 2016, which is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed December 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed November 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8,921,396), filed August 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010. The '028 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

89. I understand that the asserted claims of the '209 patent are claims 1–11, where claim 1 is independent. The claims are as follows:

> 1. A process for preparing a pharmaceutical composition, the process comprising: dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-l-ylmethyl)benzoyl)]methylamino}-ethyl)piperidin-4-yl ester in a solvent to form a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.
>
> 2. The process of claim 1, further comprising combining the solution with an aqueous pharmaceutical carrier.
>
> 3. The process of claim 1, wherein the solvent is an aqueous pharmaceutical carrier.
>
> 4. The process of claim 1, wherein the crystalline freebase is further characterized by five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
>
> 5. The process of claim 1, wherein the crystalline freebase is further characterized by a powder x-ray diffraction pattern having peak positions in accordance with the peak positions shown in FIG. 1.
>
> 6. The process of claim 1, wherein the crystalline freebase is further characterized by a melting point of about 125° C.

7. The process of claim 1, wherein the crystalline freebase is further characterized by a differential scanning calorimetry thermogram in accordance with that shown in FIG. 4.

8. The process of claim 1, wherein the pharmaceutical composition is isotonic.

9. The process of claim 1, wherein the pharmaceutical composition has a pH of about 4-6.

10. The process of claim 1, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.

11. The process of claim 1, wherein the pharmaceutical composition contains about 0.05 µg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-l-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester.

90. The '567 application was filed on June 15, 2022. The claims were rejected in two rounds of non-final rejections, and the applicant responded in both instances. The examiner allowed the claims and explained "crystalline freebase form of [revefenacin] having a powder X-ray diffraction pattern comprising" specific diffraction peaks, "in accordance with that shown in Fig. 1 (form III as defined in Applicant's specification) is not taught or suggested by the closest prior art." Ex. L, '209 patent prosecution history, Apr. 5, 2023 Notice of Allowance at PLTF-YUP-00004057-PLTF-YUP-00004058.

G. THE '948 PATENT

91. The '948 Patent, entitled "Crystalline freebase forms of a biphenyl compound," issued on July 4, 2023, and is assigned on its face to Theravance Biopharma R&D IP, LLC. The named inventor is Grahame Woollam. Ex. C, '948 patent at Cover. The '948 patent was filed as U.S. Application No. 17/301,820 ("the '820 application") on April 15, 2021. The '948 patent is a continuation of U.S. Application No. 16/715,255 (issued as the '289 patent), filed Dec. 16, 2019, which is a continuation of U.S. Application No. 16/130,079 (issued as the '081 patent), filed, Sept. 13, 2018, which is a continuation of U.S. Application No. 15/677,264 (issued as the '013 patent), filed on August 15, 2017, which is a continuation of U.S. Application No. 15/206,877 (issued as

the ’028 patent), filed July 11, 2016, which is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed Dec. 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed Nov. 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8,921,396), filed Aug. 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010.

92. I understand that the ’948 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

93. The ’948 Patent contains twenty-one claims, all of which I understand are currently asserted against the Defendants, and of which claims 1, 4, 6, and 15–21 are independent. The claims are as follows:

> 1. A pharmaceutical composition useful for treating chronic obstructive pulmonary disease in a human patient, produced by the following step: dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{1[4-(4-carbamoylpiperidin-1-ylmethyl) benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solvent to form a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and wherein the pharmaceutical composition has a pH of about 5.
>
> 2. The pharmaceutical composition of claim 1, wherein the solution is combined with an aqueous pharmaceutical carrier.
>
> 3. The pharmaceutical composition of claim 1, wherein the solvent is an aqueous pharmaceutical carrier.
>
> 4. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1; and wherein crystalline freebase is dissolved in a solvent

and then combined with the pharmaceutically acceptable carrier; and wherein the pharmaceutical composition has a pH of about 5.

5. The pharmaceutical composition of claim 4, wherein the crystalline freebase is further characterized by a melting point of about 125° C.

6. A pharmaceutical composition comprising: a dissolved crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl) benzoyl]methylamino}-ethyl)piperidin-4-yl ester and an aqueous pharmaceutical carrier; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and wherein the pharmaceutical composition has a pH of about 5.

7. The pharmaceutical composition of claim 4, wherein the pharmaceutical composition is isotonic.

8. The pharmaceutical composition of claim 4, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.

9. The pharmaceutical composition of claim 4, wherein the pharmaceutical composition contains about 0.05m/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino }ethyl)piperidin-4-yl ester.

10. The pharmaceutical composition of claim 4, wherein the pharmaceutical composition is administered by inhalation using a nebulizer inhaler.

11. The pharmaceutical composition of claim 6, wherein the pharmaceutical composition is isotonic.

12. The pharmaceutical composition of claim 6, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.

13. The pharmaceutical composition of claim 6, wherein the pharmaceutical composition contains about 0.05 μm/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester.

14. The pharmaceutical composition of claim 6, wherein the pharmaceutical composition is administered by inhalation using a nebulizer inhaler.

15. A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

16. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is in solution with the pharmaceutically acceptable carrier.

17. A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C.

18. A pharmaceutical composition comprising a solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a melting point of about 125° C.

19. A pharmaceutical composition comprising a solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

20. A solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a melting point of about 125° C.

21. A solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

94. The ’820 application was filed on April 15, 2021. The claims were rejected in a non-final rejection, and the applicant responded. The examiner allowed the claims and explained “the claimed pharmaceutical composition comprising crystalline freebase of [revefenacin] having a powder X-ray diffraction pattern comprising” specific diffraction peaks “in accordance with that

shown in Fig. 1 (form III as defined in Applicant's specification), having a melting point of about 125 °C is not taught or suggested by the closest prior art." Ex. D, '948 patent prosecution history, Apr. 12, 2023 Notice of Allowance at PLTF-YUP-00004498-PLTF-YUP-00004499.

H. THE '898 PATENT

95. The '898 patent, entitled "Crystalline freebase forms of a biphenyl compound," issued on January 2, 2024, and is assigned on its face to Theravance Biopharma R&D IP, LLC. The named inventor is Grahame Woollam. Ex. E, '898 patent at Cover. The '898 patent was filed on May 19, 2023 as U.S. Application No. 18/199,812 ("the '812 application"). The '898 patent is a continuation of U.S. Application No. 17/301,820 (issued as the '948 patent), filed April 15, 2021, which is a continuation of U.S. Application No. 16/715,255 (issued as the '289 patent), filed Dec. 16, 2019, which is a continuation of U.S. Application No. 16/130,079 (issued as the '081 patent), filed, Sept. 13, 2018, which is a continuation of U.S. Application No. 15/677,264 (issued as the '013 patent), filed on August 15, 2017, which is a continuation of U.S. Application No. 15/206,877 (issued as the '028 patent), filed July 11, 2016, which is a continuation of U.S. Application No. 14/955,515 (issued as U.S. Patent No. 9,415,041), filed Dec. 1, 2015, which is a continuation of U.S. Application No. 14/547,455 (issued as U.S. Patent No. 9,226,896), filed Nov. 19, 2014, which is a continuation of U.S. Application No. 13/973,174 (issued as U.S. Patent No. 8.921,396), filed Aug. 22, 2013, which is a division of U.S. Application No. 12/835,964 (issued as U.S. Patent No. 8,541,451), filed July 14, 2010.

96. I understand that the '898 patent claims priority to U.S. Provisional Application No. 61/225,803, filed on July 15, 2009.

97. I understand that the asserted claims of the '898 patent are claims 1–27, where claims 1, 9, and 12 are independent. The claims are as follows:

1. A method for treating chronic obstructive pulmonary disease in a human patient, the method comprising:

administering a pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester;

wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1 and 20.2±0.1.

2. The method of claim 1, wherein the crystalline freebase is further characterized by a melting point of about 125° C.

3. The method of claim 1, wherein the crystalline freebase is further characterized by a differential scanning calorimetry thermograph which shows an onset of endothermic heat flow at about 123° C.

4. The method of claim 1, wherein the pharmaceutical composition is isotonic.

5. The method of claim 1, wherein the pharmaceutical composition has a pH of about 4-6

6. The method of claim 1, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.

7. The method of claim 1, wherein the pharmaceutical composition contains about 0.05 μg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester.

8. The method of claim 1, wherein the pharmaceutical composition is administered to the human patient using a nebulizer.

9. A crystalline form of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester, wherein the crystalline form is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 11.4±0.1 and 11.6±0.1.

10. The crystalline form of claim 9, wherein the crystalline form is characterized by a DSC thermograph which shows an onset of endothermic heat flow at about 123° C.

11. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline form of claim 9.

12. A crystalline form of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester, wherein the crystalline form is characterized by a differential scanning calorimetry thermograph which shows an onset of endothermic heat flow at about 123° C.

13. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline form of claim 12.

14. A method for treating chronic obstructive pulmonary disease in a human patient, the method comprising:

administering the pharmaceutical composition of claim 11.

15. The method of claim 14, wherein the pharmaceutical composition is isotonic.

16. The method of claim 14, wherein the pharmaceutical composition has a pH of about 4-6.

17. The method of claim 14, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.

18. The method of claim 14, wherein the pharmaceutical composition contains about 0.05 μg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester.

19. The method of claim 14, wherein the pharmaceutical composition is administered to the human patient using a nebulizer.

20. A method for treating chronic obstructive pulmonary disease in a human patient, the method comprising:

administering the pharmaceutical composition of claim 13.

21. The method of claim 20, wherein the pharmaceutical composition is isotonic.

22. The method of claim 20, wherein the pharmaceutical composition has a pH of about 4-6.

23. The method of claim 20, wherein the pharmaceutical composition is buffered with citrate buffer to a pH of about 5.

24. The method of claim 20, wherein the pharmaceutical composition contains about 0.05 μg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester.

25. The method of claim 20, wherein the pharmaceutical composition is administered to the human patient using a nebulizer.

26. A pharmaceutical composition prepared from a crystalline form of claim 9 by adding a pharmaceutically acceptable carrier.

27. A pharmaceutical composition prepared from a crystalline form of claim 12 by adding a pharmaceutically acceptable carrier.

98. The ’812 application was filed on May 19, 2023. The claims were rejected in a non-final rejection, and the applicant responded. The examiner allowed the claims and explained “the claimed pharmaceutical composition and methods of treating COPD comprising crystalline

freebase form of [revefenacin] having a powder X-ray diffraction pattern comprising" specific diffraction peaks, "having a melting point of about 125 °C, and a DSC thermograph showing an onset of endothermic heat flow at about 123 °C, disclosed in Applicant's specification as polymorph form III, is not taught or suggested by the closest prior art." Ex. F, '898 patent prosecution history, Nov. 15, 2023 Notice of Allowance at PLTF-YUP-00487412-PLTF-YUP-00487413.

VIII. CLAIM TERMS

A. "CRYSTALLINE FREEBASE"; "CRYSTALLINE COMPOUND"; "CRYSTALLINE FORM"

99. I understand that the parties have proposed different constructions for "crystalline freebase," "crystalline compound," and "crystalline form." I understand that Defendants have proposed constructions for each of these terms. I further understand that Plaintiffs believe that no construction is required of the terms "crystalline freebase," "crystalline compound," and "crystalline form", but that to the extent that a construction is required, they have provided the following constructions:

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"crystalline freebase" '451 patent, claims 1 and 5 '028 patent, claims 1-6 '013 patent, claims 1-5 '081 patent, claims 1-4 '289 patent, claims 1-5 '209 patent, claims 1 and 4-7 '948 patent, claims 1, 4-6, and 15-21 '898 patent, claims 1-3	No construction required. To the extent Defendants maintain that construction is required, this term should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., crystalline freebase, a freebase substance having a regular repeating pattern of molecules that extends over long range in three dimensions.	a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"crystalline compound" '451 patent, claims 2-3 and 6	No construction required. To the extent Defendants maintain that construction is required, this term should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art in the context of each claim—i.e., as part of the phrases "crystalline compound of claim 1" and "crystalline compound of claim 5."	a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions
"crystalline form" '898 patent, claims 9-13 and 26-27	No construction required. To the extent Defendants maintain that construction is required, this term should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., crystalline form, solid form having a repeating pattern of molecules that extends over long range in three dimensions.	a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions

100. I have considered the constructions that are proposed by the parties for "crystalline freebase," "crystalline compound," and "crystalline form," and in my opinion, based on my experience and the evidence I have considered, a POSA would understand these terms to mean a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions. My opinion is based on intrinsic and extrinsic evidence and discussed in detail below.

1. "CRYSTALLINE FREEBASE"

101. For the term "crystalline freebase," the only dispute appears to be whether "crystalline freebase" is solid. The differences in the constructions are presented below in red text, with the most substantive difference highlighted in yellow.

- **Plaintiffs' Construction:** a freebase substance having a regular repeating pattern of molecules that extends over long range in three dimensions
- **Defendants' Construction:** a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions

102. Both parties appear to agree that the freebase has a regular repeating pattern of molecules that extends over long range in three dimensions. This is consistent with the understanding of a POSA because a crystalline freebase will have a regular repeating pattern, and this pattern extends in three dimensions over a long range, as I discussed above in paragraph 43.

103. I understand that claim terms should be considered in the context of the entire patent claim in which they appear, as well as in the context of the other claims, the specification, and the prosecution history of the patent.

104. I understand that Plaintiffs believe that "crystalline freebase" should be given its plain and ordinary meaning. A POSA would understand that the plain and ordinary meaning is that "crystalline freebase" must be a solid.

105. The specifications[7] and the claim terms themselves indicate that the crystalline freebase is characterized by certain XRPD peaks. As I explain above, XRPD, as the very name implies (x-ray ***powder*** diffraction), is a method of characterizing solids. (*See above* at Section VI.C.1). A POSA would understand that an XRPD pattern with sharp, distinct peaks is indicative

[7] I understand that the Polymorph Patents share a common specification. For ease of reference throughout my declaration, I will refer to the specifications of the '451 patent, '013 patent, and '948 patent but the references equally apply to the specifications of the other Polymorph Patents.

of a crystalline solid. For example, claims 1 and 5 of the '451 patent, Ex. A; claims 1-6 of the '028 patent, Ex. G; claims 1-3 of the '013 patent, Ex. O; claims 1-3 of the '289 patent, Ex. I; claims 1, and 4-5 of the '209 patent, Ex. K; claims 1, 4, 6, 15-17, 19, and 21 of the '948 patent, Ex. C; and claim 1 of the '898 patent, Ex. E, explain that the "crystalline freebase" is characterized by XRPD having diffraction peaks at certain 2θ values. Similarly, claim 9 of the '898 patent, Ex. E, explains that the "crystalline form" is characterized by XRPD having diffraction peaks at certain 2θ values. Likewise, claims 2, 3, and 6 of the '451 patent, Ex. A, explain that the "crystalline compound" is characterized by XRPD having diffraction peaks at certain 2θ values. Similarly, claims 1-6 of the '028 patent, Ex. G, explain that the "crystalline compound" is characterized by XRPD having diffraction peaks at certain 2θ values. Turning to the specification, Example 5 discusses XRPD, patterns of crystalline forms III and IV, which are the crystalline forms described in the Polymorph Patents. Ex. A, '451 patent at Example 5. Example 5 explains that the sample "was prepared by mounting a few milligrams of sample on a Silicon wafer [] plate, resulting in a thin layer of powder." Ex. A, '451 patent at 22:39-49. Example 5 further provides "[c]haracteristic peak positions" of the specific forms of revefenacin and representative XRPD patterns for each form. Ex. A,'451 patent at 22:50-23:23.

106. The specifications and claim terms also indicate that the crystalline freebase is characterized by specific melting points. A POSA would understand that melting point is indicative that the substance is a solid under ambient conditions of temperature and pressure because solids, as opposed to liquids or gases, have characteristic melting points at the temperature at which the substance transforms from a solid phase to a liquid phase. For example, claims 1 and 5 of the '451 patent, Ex. A; claim 4 of the '013 patent, Ex. O; claims 1 and 3 of the '081 patent, Ex. M; claim 4 of the '289 patent, Ex. I; claim 6 of the '209 patent, Ex. K; claim 5, 17–-18, and

20 of the '948 patent, Ex. C; and claim 2 of the '898 patent, Ex. E, further explain that "crystalline freebase" is characterized by a certain melting point. A POSA would further understand that melting point is indicative of a solid form because solids are characterized by melting points. Additionally, Example 6 describes TGA during which the sample melts. Ex. A, '451 patent at 23:49-59. Melting is also a process inherent to solids.

107. In the state of the art section in the specifications, the patentee emphasizes two "highly desirable" characteristics that indicate the crystalline freebase is a solid: melting points and ability to be micronized.

> When preparing pharmaceutical compositions and formulations for use in such devices, it is ***highly desirable to have a crystalline form of the therapeutic agent*** that is neither hygroscopic nor deliquescent and which has a ***relatively high melting point thereby allowing the material to be micronized*** without significant decomposition. Although crystalline freebase forms of the compound of formula I have been reported in U.S. Patent Publication No. 2007 /0112027 to Axt et al. as Form I and Form II, the crystalline freebase forms of the present invention have different and particularly useful properties, ***including higher melting points***.

Ex. A, '451 patent at 1:61-2:5. Additionally, the patentee highlights here that one of the distinguishing features of the crystalline forms described in the Polymorph Patents is the melting point. Ex. A, '451 patent at 1:61-2:5. A POSA would understand that the crystalline form must be solid to have a "relatively higher melting point" and "to be micronized".

108. The specifications additionally mention micronization in Example 8, which further supports that the crystalline freebase is solid. Ex. A, '451 patent at Example 8, 24:16-47. Example 8 describes micronization of crystalline freebase form III of revefenacin where "the particle size [was] determined buy [sic] laser light diffraction," and provides the particle size of micronized material in μm. Ex. A, '451 patent at 24:20-30. Example 8 further reports that "Micronization yielded particles in the respirable size range." Ex. A, '451 patent at 24:33-34. Example 8

additionally reports DSC thermograms for the crystalline freebase Form III that report "a sharp melt at 125°C" "with no evidence of amorphous content." Ex. A, '451 patent at 24:40-47.

109. Additionally, the specifications of the Polymorph Patents explain various methods of synthesis, which further support that the crystalline freebase is solid. The specifications explain that the crystalline freebase forms of the invention can be produced via several methods and that "the crystalline content as well as the habit of the crystals (size and shape) may vary[.]" Ex. A, '451 patent at 5:44-53. A POSA would understand that reference to the "habit of the crystals" means that the crystalline freebase is solid. The specification further explains that "[u]pon completion, of any of the foregoing crystallizations, the crystalline compounds can be isolated from the reaction mixture by any conventional means such as precipitation, concentration, centrifugation and the like[,]" which also supports that crystalline is as solid as solids are subject to, for instance, precipitation and centrifugation. Ex. A, '451 patent at 6:8-12.

110. Further, the synthesis methods described in the specifications indicate the crystalline freebase, including Form III and Form IV, which involved solvent-mediated methods as described above, is solid. With respect to Form III of revefenacin, for example, the specifications state that Form III crystalline freebase is prepared by contacting revefenacin with acetonitrile, at the end of which reaction "[t]he ***solids*** are then isolated by vacuum filtration and dried." Ex. A, '451 patent at 6:35-44 (emphasis added). "In another embodiment, the Form III crystalline freebase is prepared using a seed crystal of the Form III crystalline freebase… [and the resulting] suspension is stirred [and] collected by ***filtration***. The resulting filter cake is washed with isopropyl acetate [] and the product is then ***dried*** to yield the Form III crystalline freebase." Ex. A, '451 patent at 6:45-7:6 (emphasis added). This further supports the crystalline freebase is a solid. The specification similarly teaches the preparation of Form IV of revefenacin crystalline

freebase where the resulting Form IV "***solids*** are [] isolated by vacuum filtration and dried." Ex. A, '451 patent at 7:10-24.

111. The specifications further provide several examples which support that crystalline freebases are solid. For example, Preparation 1 of the specifications provides a method of preparing the crystalline revefenacin where the resultant "***solids*** were dried in an oven for about 48 hours to yield the title compound[.]" Ex. A, '451 patent at 20:10-24 (emphasis added). Example 1 describes a crystalline revefenacin preparation where the resultant "***solids*** were isolated by vacuum filtration using a sinter funnel, then placed in the piston dryer at 40 °C under full vacuum for 15.5 hours, to yield 76.85 mg of the title crystalline compound." Ex. A, '451 patent at 20:26-45 (emphasis added). Further, Example 2 explains that the resultant "***solids*** were collected by ***filtration*** [and] the product was ***dried*** [] to yield the title crystalline compound." Ex. A, '451 patent at 21:10-17 (emphasis added). The additional revefenacin synthesis Examples 3 and 4 also describe resultant solid products, including in the form of "white powder" and which undergo drying as the last step in their synthesis. Ex. A, '451 patent at 21:20-22:34.

112. A POSA would understand that the specification claims support that "crystalline freebase," as well as "crystalline compound" and "crystalline form," refer to a solid and should be construed as a solid freebase with a regular repeating pattern of freebase molecules that extends over long-range in three-dimensions.

113. The prosecution histories of the Polymorph Patents further support that "crystalline freebase," "crystalline compound," and "crystalline form" refer to a solid because the applicant and the examiner repeatedly emphasized XRPD and melting points as characteristics that were different from the prior art. For example, during the prosecution of the '451 patent, the applicant emphasized melting point as a point of novelty to distinguish from the other known forms of

revefenacin. In a non-final rejection, the examiner observed that "[i]t would have been prima facie obvious that crystalline freebase form III [of revefenacin] [] would have been characterized by a [] powder x-ray diffraction peaks [and] a melting point of about 125 °C[,]" both of which are properties inherent to solids. Ex. B, '451 patent prosecution history Nov. 29, 2012 Non-Final Rejection at PLTF-YUP-00000545. In a response, the applicant argued that the claimed revefenacin Forms III and IV are different from Forms I and II in the prior art. Ex. B,'451 patent prosecution history Feb. 27, 2013 Response to Non-Final Rejection at PLTF-YUP00000655-PLTF-YUP-00000657. The applicant also emphasized the importance of "higher melting points" as "particularly useful properties," and stressed the importance of the claimed invention's "different PXRD profiles AND different melting points[.]" *Id.* at PLTF-YUP-00000657-PLTF-YUP-00000658 (underlining in original). Those higher melting points, the applicant claimed, also "allow for the material to be processed, for example, micronized, without significant decomposition." *Id.* at PLTF-YUP-00000660. In the notice of allowance, the examiner noted the prior art had melting points "considerably lower than the melting points of crystalline forms III and IV as cited in the instant claims." Ex. B, '451 patent prosecution history, Notice of Allowance at PLTF-YUP-00000676. The examiner further noted that prior art "does not disclose that the compound has x-ray diffraction peaks of 2θ values" of certain values "or the melting points of Form III and IV as cited in the instant claims." *Id.* at PLTF-YUP-00000675.

114. In another example, during the prosecution of the '081 patent, the applicant asserted that the claimed revefenacin "Form III is prepared under different conditions and it has significantly higher melting point compared to [prior art] Forms I and II." Ex. N, '081 patent prosecution history, Jul. 10, 2019 Response to Office Action at PLTF-YUP-00002542. The applicant also explained that the prior art "did not find the presently claimed crystalline freebase

form having a highly desirable, significantly higher melting point" and that "[t]he presently claimed crystalline freebase form is prepared under different conditions than those used [in the prior art] and it has different physical properties, i.e., a significantly higher melting point." Ex. N,'081 patent prosecution history, Jul. 10, 2019 Response to Office Action at PLTF-YUP-00002543, PLTF-YUP-00002546. In the notice of allowance, the examiner noted that the claimed invention "having a melting point of about 125 ℃ [] is not taught or suggested by" the prior art. Ex. N, '081 patent prosecution history, September 17, 2019 Notice of Allowance at PLTF-YUP-00002566. The examiner further noted that the prior art "polymorphs are prepared by different processes (different solvents) from the instantly claimed crystalline polymorph, are characterized by different PXRD patterns, and the polymorphs of [prior art] have [different] melting peaks[.]" Ex. N,'081 patent prosecution history, September 17, 2019 Notice of Allowance at PLTF-YUP-00002566, PLTF-YUP-00002568. The examiner also noted that the claimed crystalline form has a melting point "not taught or suggested by the prior art." Ex. N, '081 patent prosecution history, September 17, 2019 Notice of Allowance at PLTF-YUP-00002569.

115. In each of the notices of allowance filed in the Polymorph Patents, the examiner highlighted either melting point or XRPD, as the point of distinction from prior art. Ex. B, '451 patent prosecution history, May 28, 2019 Notice of Allowance at PLTF-YUP-00000675-PLTF-YUP-00000676; Ex. P, '013 patent prosecution history, June 15, 2018 Notice of Allowance at PLTF-YUP-00002218-PLTF-YUP-00002219; Ex. H, '028 patent prosecution history, May 25, 2017 Notice of Allowance at PLTF-YUP-00001852-PLTF-YUP-00001853; Ex. N, '081 patent prosecution history, September 17, 2019 Notice of Allowance at PLTF-YUP-00002566-PLTF-YUP-00002568; Ex. L, '209 patent prosecution history, April 5, 2023 Notice of Allowance at PLTF-YUP-00004057-PLTF-YUP-00004058; Ex. J, '289 patent prosecution history, January 25,

2021 Notice of Allowance at PLTF-YUP-00002877-PLTF-YUP-00002878; Ex. F, '898 patent prosecution history, November 15, 2023 Notice of Allowance at PLTF-YUP-00487412-PLTF-YUP-00487413; Ex. D, '948 patent prosecution history, April 12, 2023 Notice of Allowance at PLTF-YUP-00004498-PLTF-YUP-00004499. Therefore, the prosecution history of the Polymorph Patents further supports that crystalline freebase, crystalline compound, and crystalline form necessarily refer to a solid.

116. The extrinsic support further is consistent with the construction of "crystalline freebase" to mean "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." These are detailed below:

Reference	**Definition**
Myerson & Ginde 2002 (Ex. AB) at DEFSREV0007876	"*Crystals* are solids in which the atoms are arranged in a periodic repeating pattern that extends in three dimensions. While all crystals are solids, not all solids are crystals." "Crystals are solids in which the atoms are arranged in a three-dimensional repeating periodic structure."
Byrn 1999 (Ex. Z) at DEFSREV0000700	"One definition of a crystal is that of a solid in which the component molecules are arranged, or 'packed,' in a highly ordered fashion."
Cullity & Stock 2001 (Ex. Y) at DEFSREV0007766	"A crystal may be defined as a solid composed of atoms, ions or molecules arranged in a pattern periodic in three dimensions."
Ohannesian 2002 (Ex. AA) at DEFSREV0008162	"The solid forms attained by organic compounds span a range of molecular order [] At one extreme is the amorphous state. characterized by no regular arrangement of molecules, as in a liquid. At the other is the crystalline state. In a crystal the molecules exist in fixed conformations and are packed against each other in a regular way."

117. Accordingly, a POSA would understand "crystalline freebase" to refer to "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions."

2. "CRYSTALLINE COMPOUND"

118. I understand that patents contain independent claims and dependent claims. I further understand that dependent claims must contain all of the limitations of the independent claim. I additionally understand that dependent claims are narrower in scope as compared to independent claims.

119. The term "crystalline compound" only appears in dependent asserted claims 2, 3, and 6 of the '451 patent, Ex. A. These claims are reproduced below, as well as the coinciding independent claims. I have denoted the "crystalline compound" term in the dependent claims in red and the "crystalline freebase" term in the independent claims in blue.

> 1. A crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1; designated as Form III; and having a melting point of about 125° C.
>
> > 2. The crystalline compound of claim 1, characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values selected from 6.6±0.1, 11.4±0.1, 13.1±0.1, 16.1±0.1, 17.5±0.1, 18.2±0.1, 18.6±0.1, 19.3±0.1, 19.7±0.1, 19.9±0.1, 20.2±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.
> >
> > 3. The crystalline compound of claim 1, further characterized by a powder x-ray diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in FIG. 1.
>
> 5. A crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 10.6±0.1, 15.0±0.1, 16.0±0.1, 17.3±0.1, 17.7±0.1, 20.9±0.1, 21.4±0.1, 22.6±0.1, 24.6±0.1, and 27.8±0.1; designated as Form IV; and having a melting point of about 119° C.

> 6. The crystalline compound of claim 5, further characterized by a powder x-ray diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in FIG. 2.

120. Because the term "crystalline compound" appears in dependent claims that depend from an independent claim, a POSA would understand that the "crystalline compound" of claims 2, 3, and 6 refer to the crystalline freebase of claims 1 and 5, respectively. A POSA would understand that the "crystalline compound" means the same thing as "crystalline freebase" in the context of the claims themselves.

121. Additional patents in the family of the Polymorph Patents use "crystalline compound" in the same manner. For example, claim 1 of U.S. Patent No. 9,415,041, is directed to "[a] pharmaceutical composition comprising a pharmaceutically acceptable dry powder excipient and a crystalline freebase . . ." Ex. V, U.S. Patent No. 9,415,041 at claim 1. Multiple dependent claims use "crystalline compound" to refer to the "crystalline freebase" of claim 1. Ex. V, U.S. Patent No. 9,415,041 at claim 4 ("The composition of claim 1, wherein the ***crystalline compound*** is in micronized form."); claim 11 ("The composition of claim 1, wherein the ***crystalline compound*** is characterized by a powder x-ray diffraction pattern . . ."") (emphasis added). As another example, claim 1 of U.S. Patent No. 9,226,896, is directed to "[a] pharmaceutical composition comprising a pharmaceutically acceptable propellant and a crystalline freebase . . ." Ex. W, U.S. Patent No. 9,226,896 at claim 1. Again, multiple dependent claims use "crystalline compound" to refer to the "crystalline freebase" of claim 1. Ex. W, U.S. Patent No. 9,226,896 at claim 2 ("The composition of claim 1, wherein the ***crystalline compound*** is characterized by a powder x-ray diffraction pattern . . ."); claim 5 ("The composition of claim 1, wherein the ***crystalline compound*** is in a suspension."); claim 11 ("The composition of claim 1, comprising

from about 0.01 to 5% by weight of the ***crystalline compound*** . . ."); claim 12 ("The composition of claim 1, wherein the ***crystalline compound*** is micronized."). Thus, a POSA would understand that the applicant has use similar dependent language of "crystalline compound" to refer to "crystalline freebase," just as the applicant has done in the '451 patent.

122. Accordingly, a POSA would understand "crystalline compound" to refer to "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions."

3. "CRYSTALLINE FORM"

123. Unlike "crystalline freebase," Plaintiffs have construed "crystalline form" differently than "crystalline freebase." The differences in Plaintiffs' constructions of the terms are highlighted below:

- **Plaintiffs' Construction of "crystalline freebase":** a freebase substance having a regular repeating pattern of molecules that extends over long range in three dimensions.
- **Plaintiffs' Construction of "crystalline form":** solid form having a repeating pattern of molecules that extends over long range in three dimensions

124. Here, Plaintiffs admit that crystalline form is solid. Because crystalline compound, crystalline form, and crystalline freebase mean the same thing in the context of the Polymorph Patents, the other two terms, crystalline compound and crystalline freebase, necessarily refer to a solid. In other words, Plaintiffs' construction of "crystalline form" should apply equally to "crystalline compound" and "crystalline freebase" in the context of these specific patents. There should be no dispute that "crystalline," regardless of whether it is "crystalline form," "crystalline freebase," or "crystalline compound," is a solid.

Additionally, in my opinion, Plaintiffs' constructions are inconsistent with each other. In the context of this patent, a POSA would understand that crystalline freebase and crystalline forms

refer to a solid freebase that has a **regular** repeating pattern of molecules that extends over long range in three dimensions. It is unclear why Plaintiffs have dropped "regular" with respect to the construction of "crystalline freebase." A POSA would understand that "crystalline freebase" and "crystalline form" have a regular repeating pattern, because regularity is important to define crystallinity. As explained in the Handbook of Pharmaceutical Analysis, in the crystalline state, molecules are packed in a regular way, as compared to amorphous state, which has no regular arrangement. Ex. AA, (Ohannesian 2002) at DEFSREV0008162 ("at one extreme is the amorphous state characterized by no regular arrangement of molecules, as in a liquid. At the other is the crystalline state. In a crystal the molecules exist in fixed conformations and are packed against each other in a regular way.").

125. For the term "crystalline form," the only dispute between the parties appears to be whether "crystalline form" must have a regular repeating pattern and whether the crystalline form is referring to a form or freebase, the former being a more general term. The differences in the constructions are presented below in red text, with the most substantive difference highlighted in yellow.

- **Plaintiffs' Construction:** solid form having a repeating pattern of molecules that extends over long range in three dimensions
- **Defendants' Construction:** a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions

126. A POSA would understand the term "crystalline form" to be defined in the same way as "crystalline freebase" in the context of the Polymorph Patents.

127. The patents and claims use the terms interchangeably. For example, it is clear from the claim language of the '451 patent itself that the applicant used each of the terms, crystalline freebase, crystalline form, and crystalline compound to refer to the same thing: the crystalline freebase.

Claim 13, '451 Patent	**Claim 9, '451 Patent**
13. The composition of claim 12, which further comprises an agent selected from β2 adrenergic receptor agonists, steroidal anti-inflammatory agents, phosphodiesterase-4 inhibitors, and combinations thereof; wherein **the crystalline form** and the agent are formulated together or separately.	9. The composition of claim 8, which further comprises an agent selected from β_2 adrenergic receptor agonists, steroidal anti-inflammatory agents, phosphodiesterase-4 inhibitors, and combinations thereof; wherein **the crystalline form** and the agent are formulated together or separately.
12. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and the **compound of claim 5**.	8. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and the **compound of claim 1**.
5. A **crystalline freebase** of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 10.6±0.1, 15.0±0.1, 16.0±0.1, 17.3±0.1, 17.7±0.1, 20.9±0.1, 21.4±0.1, 22.6±0.1, 24.6±0.1, and 27.8±0.1; designated as Form IV; and having a melting point of about 119° C.	1. A **crystalline freebase** of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1; designated as Form III; and having a melting point of about 125° C.

128. As illustrated above, claim 13 of the '451 patent refers to a "crystalline form" and depends from claim 12, which refers to a "compound," which in turn depends from independent claim 5, which refers to a "crystalline freebase." Ex. A, '451 patent at cls. 5, 12-13. In the same vein, claim 9 of the '451 patent refers to a "crystalline form" and depends from claim 8, which refers to a "compound," which in turn depends from claim 1, which refers to a "crystalline freebase." Ex. A, '451 patent cls. 1, 8-9. I understand that a dependent claim cannot be broader than the claim from one it depends from, and so, the references below indicate that the crystalline form, compound of claims 1 or 5, and crystalline freebase all refer to the same crystalline freebase.

129. Additional patents in the family of the Polymorph Patents use "crystalline form" in the same manner. For example, claim 1 of U.S. Patent No. 9,415,041, is directed to "[a] pharmaceutical composition comprising a pharmaceutically acceptable dry powder excipient and a crystalline freebase . . ." Ex. V, U.S. Patent No. 9,415,041 at claim 1. Dependent claim 2 uses "crystalline form" to refer to the "crystalline freebase" of claim 1. Ex. W, U.S. Patent No. 9,226,896 at claim 3 ("The composition of claim 1 . . . wherein the ***crystalline form*** and the agent are formulated together or separately."). As another example, claim 1 of U.S. Patent No. 9,226,896, is directed to "[a] pharmaceutical composition comprising a pharmaceutically acceptable propellant and a crystalline freebase . . ." Ex. W, U.S. Patent No. 9,226,896 at claim 1. Dependent claim 3 uses "crystalline form" to refer to the "crystalline freebase" of claim 1. Ex. W, U.S. Patent No. 9,226,896 at claim 3 ("The composition of claim 1 . . . wherein the ***crystalline form*** and the agent are formulated together or separately.").

130. Further, the specifications describe the "present invention" as relating to "novel crystalline forms…[and] pharmaceutical compositions comprising the crystalline compounds [and] processes and intermediates for preparing such crystalline compounds[.]" Ex. A, '451 patent at 1:14-21. Therefore, it is my opinion that a POSA will understand, in light of the claims and the specifications, that crystalline freebase, crystalline form, and crystalline compounds are used interchangeably within the patents.

131. Additionally, the prosecution history further supports the interchangeability of "crystalline freebase" and "crystalline form." For example during prosecution of the '209 patent and the '948 patent, the examiner used "crystalline freebase" and "crystalline form" as interchangeable terms, by referring to pending "crystalline freebase" claims as "identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising the same crystalline

form of the same compound[.]" Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Office Action at PLTF-YUP-00003920-PLTF-YUP-00003921; *see also, e.g.*, Ex. D, '948 patent prosecution history, Feb. 10, 2023 Non-Final Office Action at PLTF-YUP-00004325 (same); Ex. B, '948 patent Feb. 10, 2023 Non-Final Office Action at PLTF-YUP-00004324 (referring to pending "crystalline freebase" claims as "identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising the same pharmaceutically acceptable carrier and the same crystalline form of the same compound[.]"); Ex. L, '209 patent prosecution history, Aug. 22, 2022 Non-Final Rejection at PLTF-YUP-00003854-PLTF-YUP-00003855 ("The instant claims and claim of US '451 are therefore not patentably distinct because both sets of claims encompass the same crystalline form of [revefenacin] freebase[.]"); PLTF-YUP-00003859 (discussing "crystalline freebase" claims of the '028 patent: "[b]oth sets of claims therefore require the same crystalline form of the same compound."); PLTF-YUP-00003861 (discussing pending "crystalline freebase" claims: "[t]he compound recited in both sets of claims is the same crystalline form, as evidenced by the same melting point and differential scanning calorimetry thermogram[.]"); PLTF-YUP-00003864 (discussing pending "crystalline freebase" claims: "[t]he compound recited in both sets of claims is the same crystalline form, as evidenced by the same melting point, powder x-ray diffraction peaks, and differential scanning calorimetry thermogram[.]"); PLTF-YUP-00003865 (discussing pending "crystalline freebase" claims: the copending claims are drawn to a process of preparing a composition comprising dissolving the same crystalline form of the same compound in a solvent.).

132. Thus, for the reasons discussed, a POSA would understand "crystalline form" to refer to "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions."

B. "SOLUTION"

133. I understand the parties have proposed different constructions of the term "solution," which are presented below.

Disputed Term/Phrase	Plaintiffs' Proposed Construction	Defendants' Proposed Construction
"solution" '013 patent, claims 1 and 6-9 '209 patent, claims 1-2 '948 patent, claims 1-2 and 15-21	No construction required. To the extent Defendants maintain that construction is required, this term should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., solution, a homogenous mixture of molecules.	liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents

134. I have considered the constructions that are proposed by the parties for "solution," and in my opinion, based on my experience and the evidence I have considered, a POSA would understand the term "solution" to mean a "liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents."

135. The definition of solution is consistent with the intrinsic evidence in this case, and is the definition proffered by the United States Pharmacopeia, which defines solutions as "liquid preparations that contain one or more chemical substances dissolved, i.e. molecularly dispersed, in a suitable solvent or mixture of mutually miscible solvents." Ex. AE, (USP 2008) at DEFSREV0007983.

136. Plaintiffs' construction, although not scientifically inaccurate, is very broad and encompasses, for example, solid solutions. In my opinion, Defendants' construction is more consistent with the use of "solution" in the context of the patent, e.g. as a pharmaceutical

composition. Additionally, because Defendants' construction makes it clear that the chemical substance is dissolved, it is more consistent with a POSA's understanding of solution in the pharmaceutical context: a POSA would understand that a crystalline form was dissolved to form a "solution" using a liquid solvent and that the solution would therefore retain no memory of the crystalline form.

137. The term "solution" is not defined in the specification, and it generally appears in the specification in two contexts: as a pharmaceutical composition and as part of the process steps to form a crystalline freebase. Looking at how "solution" is used in the claims, in almost all of the Asserted Claims containing the term "solution," the term "pharmaceutical composition" also appears, and a POSA would understand that each of these claims refers to solution as a pharmaceutical composition. Moreover, the specification provides in the Abstract and Field of Invention that the invention relates to pharmaceutical compositions comprising the crystalline freebase or prepared from the crystalline freebase. *See, e.g.*, Ex. O, '013 patent at Abstract ("The invention also provides pharmaceutical compositions comprising the crystalline freebase or prepared using the crystalline freebases."); 1:49-58 ("The invention also relates to pharmaceutical compositions comprising the crystalline compounds or prepared from such compounds."). A POSA would understand that "solution" refers to a pharmaceutical composition, when used in the claims. The claims are below:

Patent Claim	**Claim Language**
'013 patent, claims 1 and 6-9	1. A method for preparing a pharmaceutical composition for use in a nebulizer inhaler, the method comprising dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in an aqueous pharmaceutical carrier to form an aqueous solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

	6. The method of any one of claims 1 to 5, wherein the aqueous solution is isotonic. 7. The method of any one of claims 1 to 5, wherein the aqueous solution has a pH of about 4-6. 8. The method of any one of claims 1 to 5, wherein the aqueous solution is buffered with citrate buffer to a pH of about 5. 9. The method of any one of claims 1 to 5, wherein the aqueous solution contains about 0.05 μg/mL to about 10 mg/mL of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester.
'209 patent, claims 1-2	1. A process for preparing a pharmaceutical composition, the process comprising: dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-l-ylmethyl)benzoyl)]methylamino}-ethyl)piperidin-4-yl ester in a solvent to form a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1. 2. The process of claim 1, further comprising combining the solution with an aqueous pharmaceutical carrier.
'948 patent, claims 1-2	1. A pharmaceutical composition useful for treating chronic obstructive pulmonary disease in a human patient, produced by the following step: dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{1[4-(4-carbamoylpiperidin-1-ylmethyl) benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solvent to form a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 20 values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and wherein the pharmaceutical composition has a pH of about 5. 2. The pharmaceutical composition of claim 1, wherein the solution is combined with an aqueous pharmaceutical carrier.
'948 patent, claim 15	15. A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.
'948 patent, claim 16	16. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 20 values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1,

	16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is in solution with the pharmaceutically acceptable carrier.
'948 patent, claim 17	17. A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C.
'948 patent, claim 18	18. A pharmaceutical composition comprising a solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a melting point of about 125° C.
'948 patent, claim 19	19. A pharmaceutical composition comprising a solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.
'948 patent, claim 20	20. A solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a melting point of about 125° C.
'948 patent, claim 21	21. A solution comprising a crystalline freebase of biphenyl-**2**-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

138. The intrinsic evidence supports Defendants' construction. First, the specification supports that "solution" means a liquid preparation. For example, the specification explains that "solutions" are "suitable liquid dosage forms." Ex. O, '013 patent at 14:63-66. The specification further explains that "[s]uch liquid dosage forms typically comprise the active ingredient and an inert diluent such as, for example, water or other solvents, solubilizing agents and emulsifiers[.]" Ex. O, '013 patent at 14:66-15:1.

139. Second, intrinsic evidence supports that "solution" contains a substance that is dissolved in a solvent or mixture of miscible solvents. When formulated as a solution, a POSA

would understand that the chemical substance, i.e. crystalline freebase in this case, is dissolved in a solvent. Said another way, there will be no memory of the crystalline form in the solution once it is dissolved. This is consistent with what the specification explains. Ex O, '013 patent at 10:67-11:4 ("A crystalline freebase of the invention is typically administered to a patient in the form of a pharmaceutical composition or formulation. . . . However, it will be understood by those skilled in the art that, ***once a crystalline freebase of the invention has been formulated, it may no longer be in crystalline form, i.e., the crystalline freebase may be dissolved in a suitable carrier***." (emphasis added)). A POSA would understand that once formulated as a solution, the crystalline freebase would be dissolved, leaving no memory of the crystalline form.

140. Further examples in the specification support that a substance would be dissolved in a solvent. For example, the specification explains, "Accordingly, when formulated for use in a nebulized inhaler, the crystalline freebase active agent is typically ***dissolved in a suitable carrier*** to form a solution." Ex. O, '013 patent at 12:12-15 (emphasis added). In this example, a POSA would understand that the crystalline freebase is no longer present in the solution. In another example providing an "exemplary aqueous aerosol formulation for administration by nebulizer," the "pharmaceutical composition is prepared by ***dissolving*** 0.5 mg of a crystalline freebase of the invention (active agent) ***in 1 mL of a 0.9% sodium chloride solution acidified with citric acid***. ***The mixture is stirred and sonicated until the active agent is dissolved***. The pH of the solution is adjusted to a value in the range of from 3 to 8 (typically about 5) by the slow addition of NaOH." Ex. O, '013 patent at 19:15-24 (emphasis added). In my opinion, this example additionally makes it clear that solution is a liquid preparation where the crystalline freebase is dissolved in a solvent. The process to create the solution includes stirring and sonicating until the active agent, a crystalline freebase, is no longer present.

141. The specification further provides that the crystalline freebase of revefenacin has "good solubility" and explains that "crystalline freebase Form III solutions are stable in pH 4 and pH 6 buffers for up to 7 days at 50° C. or exposed to light. The solutions are stable in water and saline for 7 days at room temperature, protected from light." Ex. O, '013 patent at 25:39-52 (Example 10). A POSA would understand this to mean that the crystalline freebase is dissolved in solution, with good stability.

142. Additionally, when "solution" is used as part of the process to create the crystalline freebase, it is also clear that the chemical substance is dissolved in the solution. *See, e.g.*, Ex. O, '013 patent at 6:65-7:7 ("dissolving the diphosphate salt of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino }ethyl)piperidin-4-yl ester in isopropyl acetate and water to form a solution"); 7:18-22 ("clear solution"); 7:32-37 ("dissolving the crystalline freebase Form III in acetonitrile to form a solution"). For example, in Example 3, the specification explains "[t]he ***suspension*** was warmed 82±3° C., and held at this temperature ***until complete dissolution was observed***. The ***solution*** was then clarified into the crystallizer vessel, followed by rinsing with toluene (373 µL)." Ex. O, '013 patent at 21:63-66. Here, it is clear that a solution is what resulted after the suspension was warmed and the particles were completely dissolved.

143. Thus, in context of the patent specification itself, a POSA would understand that the chemical substance would be dissolved when in solution.

144. The prosecution history also supports that "solution" means that the substance "is dissolved in a solvent." In a non-final rejection issued by the examiner during the prosecution of the '209 patent, the examiner rejected certain claims of the '209 patent based on statutory double patenting. Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Rejection at PLTF-

YUP-00003918-PLTF-YUP-00003921. I understand that statutory double patenting means that the claims would be directed to the same invention. I understand the examiner interpreted certain claims of the '209 patent to be the same invention as pending claims in the application underlying the '948 patent (U.S. Application 17301820). Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Rejection at PLTF-YUP-00003918-PLTF-YUP-00003921. These claims are below:

'209 Patent	**'948 Patent**	**Examiner Comments**
32. A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester that is dissolved in a solvent; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.	62. (issued as claim 15) A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.	The claims are identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising the same crystalline form of the same compound in ***solution, i.e. dissolved in a solvent***.
34. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 20 values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1,	63. (issued as claim 16) A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 20 values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1,	These claims are identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising a pharmaceutically acceptable carrier and the same crystalline form of the same compound ***dissolved in a pharmaceutically acceptable carrier, i.e. in solution with a pharmaceutically acceptable carrier***.

19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is dissolved in the pharmaceutically acceptable carrier.	18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is in solution with the pharmaceutically acceptable carrier.	
36. A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C, dissolved in a carrier.	64. (issued as claim 17) A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C.	These claims are identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising a ***solution of the same crystalline form of the same compound, i.e. dissolved in a carrier***.

Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Rejection at PLTF-YUP-00003918-PLTF-YUP-00003921.

145. Based on the non-final rejection, the examiner defined solution to be identical to being "dissolved in" a carrier or solvent. In response, the applicant cancelled the pending claims 32, 34, and 36 in the '209 patent. (Mar. 23, 2023 Reply to Non-Final Rejection at 4). I understand the same rejection was also issued in the '948 patent. Ex. D, '948 patent prosecution history, Feb. 10, 2023 Non-Final Rejection at PLTF-YUP-00004323-PLTF-YUP-00004325.

146. In addition to the United States Pharmacopeia's definition of "solution," Defendants' definition of solution is consistent with other pharmaceutical textbooks. For example, Remington's defines solution with a very similar definition as the United States Pharmacopeia: "a liquid preparation that contains one or more soluble chemical substances dissolved in water." Ex. U (Remington 2000) at DEFSREV0007916.

147. Thus, for reasons discussed above, intrinsic and extrinsic evidence supports that that solution means a liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents.

C. "DISSOLVING"

148. I understand the parties have proposed different constructions of the term "dissolving," which are presented below.

Disputed Term/Phrase	Plaintiffs' Proposed Construction	Defendants' Proposed Construction
"dissolving" '013 patent, claim 1 '289 patent, claim 1 '209 patent, claim 1 '948 patent, claim 1	No construction required. To the extent Defendants maintain that construction is required, this term should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., dissolving, molecularly homogenously dispersing.	molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form

149. I have considered the constructions that are proposed by the parties for "dissolving," and in my opinion, based on my experience and the evidence I have considered, a POSA would understand the term "dissolving" to mean molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form.

150. Plaintiffs and Defendants both appear to agree that dissolving means molecularly dispersing, but Defendants' proposed construction adds further clarity that the chemical substance is no longer in crystalline form after the process of dissolving occurs.

151. Defendants' construction is consistent with the specification, which expressly provides that the chemical substance is no longer in crystalline form when it is dissolved. The

specifications of the Polymorph Patents explain the following: "However, it will be understood by those skilled in the art that, once a crystalline freebase of the invention has been formulated, ***it may no longer be in crystalline form, i.e., the crystalline freebase may be dissolved in a suitable carrier***." Ex. A, '451 patent at 10:42-46 (emphasis added). Thus, reading this passage, a POSA would understand that when the crystalline freebase is dissolved in a suitable carrier, it is no longer in crystalline form.

152. Turning to the claims themselves, it is clear from the claim language that the crystalline freebase is no longer in crystalline form after being dissolved in a liquid because it forms a solution after being dissolved in an aqueous pharmaceutical carrier or a solvent. A POSA would understand that the crystalline freebase is dissolved to form a solution. A POSA would further understand that each of the claims are directed to pharmaceutical compositions, methods for preparing pharmaceutical compositions, and methods for treating with a pharmaceutical composition.

Patent Claim	**Claim Language**
'013 patent, claim 1	A method for preparing a pharmaceutical composition for use in a nebulizer inhaler, the method comprising dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in an aqueous pharmaceutical carrier to form an aqueous solution . . .
'289 patent, claim 1	A method for treating chronic obstructive pulmonary disease in a human patient, the method comprising: (a) preparing a pharmaceutical composition by dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in an aqueous pharmaceutical carrier; . . .
'209 patent, claim 1	A process for preparing a pharmaceutical composition, the process comprising: dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-l-ylmethyl)benzoyl)]methylamino}-ethyl)piperidin-4-yl ester in a solvent to form a solution . . .
'948 patent, claim 1	A pharmaceutical composition useful for treating chronic obstructive pulmonary disease in a human patient, produced by the following step:

	dissolving a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{1[4-(4-carbamoylpiperidin-1-ylmethyl) benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solvent to form a solution

153. As another example, the specification explains, "Accordingly, when formulated for use in a nebulized inhaler, the crystalline freebase active agent is typically ***dissolved in a suitable carrier*** to form a solution." Ex. O, '013 patent at 12:12-15 (emphasis added). In this example, a POSA would understand that the crystalline freebase is dissolved in a liquid carrier to form a solution.

154. In another example providing an "exemplary aqueous aerosol formulation for administration by nebulizer," the "pharmaceutical composition is prepared by ***dissolving*** 0.5 mg of a crystalline freebase of the invention (active agent) ***in 1 mL of a 0.9% sodium chloride solution acidified with citric acid***. ***The mixture is stirred and sonicated until the active agent is dissolved***. The pH of the solution is adjusted to a value in the range of from 3 to 8 (typically about 5) by the slow addition of NaOH." Ex. O, '013 patent at 19:15-24 (emphasis added). A POSA would understand that the crystalline freebase active agent is dissolved in the liquid, i.e. sodium chloride solution acidified with citric acid.

155. The term "dissolving" appears several times in the specification in other contexts, including the process of preparing the freebase. *See, e.g.*, Ex. O, '013 patent at 6:65-7:5 ("dissolving the diphosphate salt of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl) benzoyl]methylamino }ethyl)piperidin-4-yl ester in isopropyl acetate and water to form a solution"); 7:34-36 ("dissolving the crystalline freebase Form III in acetonitrile to form a solution"). A POSA would still understand that the crystalline freebase would no longer be in crystalline form after the dissolution process.

156. The prosecution history also supports that "dissolving" means the crystalline solid will no longer be present. In a non-final rejection issued by the examiner during the prosecution

of the '209 patent, the examiner rejected certain claims of the '209 patent based on statutory double patenting. Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Rejection at PLTF-YUP-00003918-PLTF-YUP-00003921. I understand that statutory double patenting means that the claims would be directed to the same invention. I understand the examiner interpreted certain claims of the '209 patent to be the same invention as pending claims in the application underlying the '948 patent (U.S. Application 17/301,820). Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Rejection at PLTF-YUP-00003918-PLTF-YUP-00003921. These claims are below:

'209 Patent	**'948 Patent**	**Examiner Comments**
32. A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester that is dissolved in a solvent; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.	62. (issued as claim 15) A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.	The claims are identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising the same crystalline form of the same compound in ***solution, i.e. dissolved in a solvent***.
34. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having	63. (issued as claim 16) A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further	These claims are identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising a pharmaceutically acceptable carrier and the same crystalline form of the same compound ***dissolved in a pharmaceutically***

five or more additional diffraction peaks at 20 values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is dissolved in the pharmaceutically acceptable carrier.	characterized by having five or more additional diffraction peaks at 20 values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is in solution with the pharmaceutically acceptable carrier.	***acceptable carrier, i.e. in solution with a pharmaceutically acceptable carrier***.
36. A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C, dissolved in a carrier.	64. (issued as claim 17) A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C.	These claims are identical in scope, as both sets of claims are drawn to a pharmaceutical composition comprising a ***solution of the same crystalline form of the same compound, i.e. dissolved in a carrier***.

Ex. L, '209 patent prosecution history, Dec. 28, 2022 Non-Final Rejection at PLTF-YUP-00003918-PLTF-YUP-00003921.

157. Based on the non-final rejection, the examiner defined solution to be identical to being "dissolved in" a carrier or solvent. In response, the applicant cancelled the pending claims 32, 34, and 36 in the '209 patent. Ex. L, '209 patent prosecution history, Mar. 23, 2023 Reply to Non-Final Rejection at PLTF-YUP-00004049. I understand the same rejection was also issued in the '948 patent. Ex. D, '948 patent prosecution history, Feb. 10, 2023 Non-Final Rejection at PLTF-YUP-00004323-PLTF-YUP-00004325. A POSA would understand that being dissolved in a carrier or a solvent is the same as being in solution, and in both scenarios, there would be no memory of the crystalline form.

158. The term "dissolving" appears in claims under the context of preparing a pharmaceutical composition by dissolving a crystalline freebase in an aqueous carrier or solvent to form a solution as discussed in paragraph 151. Thus, when interpreting the word "dissolving" in the context of the Polymorph Patents, a POSA would understand "dissolving" to be in the context of pharmaceutical compositions. As discussed above for the term "solution," the USP defines solutions as "liquid preparations that contain one or more chemical substances dissolved, i.e. molecularly dispersed, in a suitable solvent or mixture of mutually miscible solvents." Ex. AE, (USP 2008) at DEFSREV00007983. Thus, a POSA would understand that in this context, dissolving means molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form, which is consistent with the USP definition of solution.

159. Thus, for reasons discussed above, the intrinsic and extrinsic evidence supports that that dissolving means molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form.

D. "WHEREIN CRYSTALLINE FREEBASE IS DISSOLVED"; "WHEREIN CRYSTALLINE FREEBASE IS DISSOLVED IN A SOLVENT AND THEN COMBINED WITH THE PHARMACEUTICALLY ACCEPTABLE CARRIER"

160. I understand the parties have proposed different constructions of the terms "wherein crystalline freebase is dissolved" and "wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier," which are presented below.

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"wherein crystalline freebase is dissolved" '948 patent, claim 4	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., wherein a freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution is molecularly, homogenously dispersed.	wherein freebase is molecularly dispersed in a solvent, such that the freebase is no longer in crystalline form To the extent that the Court adopts Plaintiffs' proposed construction, claim 4 is a product-by-process claim
"wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier" '948 patent, claim 4	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., wherein a freebase substance having a repeating pattern of molecules that extends over long range in three dimensions is molecularly, homogenously dispersed in a solvent and then combined with the pharmaceutically acceptable carrier.	wherein freebase is molecularly dispersed in a solvent, such that the freebase is no longer in crystalline form, and then combined with the pharmaceutically acceptable carrier To the extent that the Court adopts Plaintiffs' proposed construction, claim 4 is a product-by-process claim

161. Both of these claim phrases include variations of two claim terms that are also being proposed for construction: "crystalline freebase" and "dissolving." As I discussed above, the proper construction of "crystalline freebase" is "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." Additionally, as discussed

above, the proper construction of "dissolving" is "molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form."

162. I have considered the constructions that are proposed by the parties for "wherein crystalline freebase is dissolved" and "wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier," and in my opinion, based on my experience and the evidence I have considered, a POSA would understand "wherein crystalline freebase is dissolved" to mean "wherein freebase is molecularly dispersed in a solvent, such that the freebase is no longer in crystalline form." The construction of the longer claim phrase, "wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier" should have the same construction, except to add that it is combined with the pharmaceutically acceptable carrier.

163. In construing the phrase "wherein crystalline freebase is dissolved," a POSA would understand that crystalline freebase is no longer in the crystalline state after being dissolved. This again, is consistent with the specification of the Polymorph Patents which explains that when a crystalline freebase is dissolved, it no longer is in crystalline form: "However, it will be understood by those skilled in the art that, once a crystalline freebase of the invention has been formulated, ***it may no longer be in crystalline form, i.e., the crystalline freebase may be dissolved in a suitable carrier***." Ex. C, '948 patent at 11:9-13 (emphasis added).

164. A POSA would also understand that in the context of the claim, the crystalline freebase will be dissolved in a solvent because claim 4 specifically explains: "wherein crystalline freebase is dissolved in a solvent." Ex. C, '948 patent at claim 4. Thus, a POSA would understand that the freebase would be molecularly dispersed in a solvent.

165. I understand that when read in view of the specification and prosecution history, claims must inform a POSA with reasonable certainty about the scope of the invention. If claims fail to inform a POSA with reasonable certainty about the scope of the invention, they are considered to be indefinite.

166. Here, when looking at claim 4 as a whole, the claim does not inform a POSA the scope of the claim with reasonable certainty because it is not clear how a pharmaceutical composition can comprise a crystalline freebase that is characterized by specific XRPD peaks, but also be in a dissolved state. As discussed in the technical background section, XRPD is a method used to characterize crystalline solids, and an XRPD pattern is an inherent characteristic, a fingerprint, of a solid form. Liquids, for example, will not be characterized by an XRPD pattern with distinct peaks. A dissolved crystalline freebase cannot be characterized by XRPD peaks. Said another way, a pharmaceutical composition cannot comprise a crystalline solid and also require the same crystalline solid to be dissolved. The claim language creates an impossibility because a POSA would not be able to conduct an XRPD analysis on dissolved crystalline freebase as the freebase molecules are molecularly dispersed in solution and would therefore not diffract X-rays.

> 4. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1; and wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier; and wherein the pharmaceutical composition has a pH of about 5.

Ex. C, '948 patent at cl. 4 (annotated).

167. Under Plaintiffs' construction, "wherein a freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution is

molecularly, homogenously dispersed," the Plaintiffs add a limitation "prior to dissolution" to indicate that the crystalline freebase is essentially a solid before being "molecularly, homogenously dispersed." A POSA would understand that Plaintiffs' construction adds a process step.

168. Additionally, Plaintiffs construction of "dissolved" to mean "molecularly, homogenously disperse" does not specify that crystalline solid is no longer present. As explained above in the context of the "dissolving" term, a POSA would understand that dissolving or dissolved means that the crystalline solid is no longer present.

169. Plaintiffs' constructions are also inconsistent for a few reasons. First, Plaintiffs' have dropped "regular" from their "crystalline freebase" construction in their proposed construction of the phrases: "wherein crystalline freebase is dissolved" and "wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier." It is unclear why Plaintiffs have dropped the word "regular" in their constructions given that Plaintiffs' construction appears to require that the "crystalline freebase" be in its crystalline state "prior to dissolution." A POSA would understand that the crystalline freebase should have a regular repeating pattern of molecules that extends over long range in three dimensions. Additionally, Plaintiffs only include the "prior to dissolution" in their construction of "wherein crystalline freebase is dissolved," but do not include this requirement in their construction of the same phrase "wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier." It is unclear whether the Plaintiffs are interpreting these two claims differently.

170. Thus, for the reasons stated above, a POSA would understand "wherein crystalline freebase is dissolved" to mean "wherein freebase is molecularly dispersed in a solvent, such that

the freebase is no longer in crystalline form" and "wherein crystalline freebase is dissolved in a solvent and then combined with the pharmaceutically acceptable carrier" to mean "wherein freebase is molecularly dispersed in a solvent, such that the freebase is no longer in crystalline form, and then combined with the pharmaceutically acceptable carrier."

E. "A DISSOLVED CRYSTALLINE FREEBASE"

171. I understand the parties have proposed different constructions of the term "a dissolved crystalline freebase" which is presented below.

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"a dissolved crystalline freebase" '948 patent, claim 6	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., a molecularly, homogenously dispersed freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution.	Indefinite To the extent this claim term is not indefinite, "a solution that is prepared by molecularly dispersing a freebase in a solvent such that the freebase is no longer in crystalline form" To the extent that the Court adopts Plaintiffs' proposed construction, claim 6 is a product-by-process claim

172. I have considered the constructions that are proposed by the parties for "a dissolved crystalline freebase" and in my opinion, based on my experience and the evidence I have considered, a POSA would not be able to understand this claim term with reasonable certainty, which I understand to mean that the claim phrase is indefinite.

173. This claim phrase again includes variations of two claim terms that are also being construed: "crystalline freebase" and "dissolving." A POSA would understand each of the claim terms, "dissolved' and "crystalline freebase," as I discuss above in Sections VIII.A.1 and VIII.C.

As I discussed above, the proper construction of "crystalline freebase" is "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." Additionally, as discussed above, the proper construction of "dissolving" is "molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form."

174. As discussed above in the context of "dissolved," a POSA would understand that crystalline freebase is no longer in the crystalline state after being dissolved. This again, is consistent with the specification of the Polymorph Patents which explains that when a crystalline freebase is dissolved, it no longer is in crystalline form: "However, it will be understood by those skilled in the art that, once a crystalline freebase of the invention has been formulated, ***it may no longer be in crystalline form, i.e., the crystalline freebase may be dissolved in a suitable carrier***." Ex. C, '948 patent at 11:9-13 (emphasis added).

175. Although a POSA would understand what each of the terms mean separately, the claim language is not clear when read in the full phrase in context of the claim. Claim 6 recites that the crystalline freebase, which I understand to refer to the "dissolved crystalline freebase," must be characterized by an XRPD pattern. A dissolved crystalline freebase cannot be characterized by an XRPD pattern because it is no longer in solid form. Thus, claim 6 does not make sense. It is unclear how a dissolved crystalline freebase can have an XRPD pattern, when it will no longer be in crystalline form when in a dissolved state.

> 6. A pharmaceutical composition comprising: a dissolved crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl) benzoyl]methylamino}-ethyl)piperidin-4-yl ester and an aqueous pharmaceutical carrier; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1; and wherein the pharmaceutical composition has a pH of about 5.

Ex. C, '948 patent at cl. 6 (annotated).

176. If a POSA must find some meaning in the claim phrase "a dissolved crystalline freebase," the only way to make sense of the phrase is to require the crystalline freebase be in a solid form before the dissolution process. Thus, a POSA would have to understand the phrase to mean "a solution that is prepared by molecularly dispersing a freebase in a solvent such that the freebase is no longer in crystalline form." A POSA would have to construe the adjectival use of "dissolved" to mean that the resulting solution is prepared by dissolving a crystalline freebase. The result of dissolving a crystalline freebase is a solution, and the solution should no longer have any crystalline solid present after the dissolution process occurs.

177. Plaintiffs offer a construction that reads in a process element to the claims, but their construction again does not make clear that the crystalline solid must be dissolved. Under Plaintiffs' construction, "a dissolved crystalline freebase," means "a molecularly, homogenously dispersed freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution." Again, Plaintiffs use the words "prior to dissolution" to signal that the process is required to form the resulting product. However, Plaintiff's construction is incomplete, as I mentioned previously above in the context of the "dissolving" term, because a POSA would understand that dissolving or dissolved means that the crystalline solid is no longer present. Under Plaintiffs' construction, it is unclear whether the crystalline freebase is still present after dissolving occurs. In the context of a pharmaceutical composition, the crystalline freebase should be dissolved, and no longer be present. One would not want a partially dissolved active ingredient in a pharmaceutical composition as this could create consistency and efficacy issues.

178. I also note that Plaintiffs' construction again drops the "regular" component from their "crystalline freebase" construction in their proposed construction of the phrase: "a dissolved crystalline freebase." It is unclear what Plaintiffs have dropped the word "regular" in their

constructions given that Plaintiffs' construction appears to require that the "crystalline freebase" be in its crystalline state "prior to dissolution."

F. "CRYSTALLINE FREEBASE OF BIPHENYL-2-YLCARBAMIC ACID 1-(2-{[4-(4-CARBAMOYLPIPERIDIN-1-YLMETHYL)BENZOYL]METHYLAMINO}-ETHYL)PIPERIDIN-4-YL ESTER IN A SOLUTION"

179. I understand the parties have proposed different constructions of the terms "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution", which is presented below.

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution" '948 patent, claim 15	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., a freebase substance having a repeating pattern of revefenacin molecules that extends over long range in three dimensions prior to dissolution in a homogenous mixture of molecules.	Indefinite To the extent that the Court adopts Plaintiffs' proposed construction, claim 15 is a product-by-process claim

180. I have considered the constructions that are proposed by the parties for "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution" and in my opinion, based on my experience and the evidence I have considered, a POSA would not be able to understand

this claim term with reasonable certainty, which I understand to mean that the claim phrase is indefinite.

181. This phrase includes two claim terms that are also being construed: "crystalline freebase" and "solution." As I discussed above, the proper construction of "crystalline freebase" is "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." The proper construction of "solution" is "liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents."

182. Although a POSA would understand "crystalline freebase" and "solution" terms separately, in the context of the claim itself and as used in the claim phrase, the phrase "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution" does not make sense to a POSA. As illustrated in claim 15 below, the crystalline freebase must be "in a solution" and also be characterized by an XRPD pattern. As discussed in the technical background section, XRPD is a method used to characterize crystalline solids, and an XRPD pattern is an inherent characteristic or fingerprint of a solid form. Liquids, for example, will not be characterized by an XRPD pattern with distinct peaks.

> 15. A pharmaceutical composition comprising: a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution; wherein the crystalline freebase is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

Ex. C, '948 patent at cl. 15 (annotated).

183. Crystalline freebase of revefenacin cannot be "in a solution," be solid, and have an XRPD pattern because this is contrary to a POSA's understanding of the term "solution." A

solution, as properly construed, is a liquid preparation where the crystalline freebase is dissolved and the crystalline solid is no longer present. This interpretation is consistent with the specification, as the specification recognizes that once a crystalline freebase is formulated, i.e. dissolved in a suitable carrier, it is no longer in crystalline form. Ex. C, '948 patent at 11:9-13. A POSA would understand that there would be no solid particulates in a solution, otherwise, that would be considered a suspension. As I discussed previously, a POSA would construe "solution" based on the definition of the USP and intrinsic evidence. The USP also defines suspension. Both definitions are presented below:

- **Solution:** liquid preparations that contain one or more chemical substances dissolved, i.e. molecularly dispersed, in a suitable solvent or mixture of mutually miscible solvents. Ex. AE (USP 2008) at DEFSREV0007983.
- **Suspension:** liquid preparations that consist of solid particles dispersed throughout a liquid phase in which the particles are not soluble. Ex. AE (USP 2008) at DEFSREV0007983.

A POSA would understand that solutions and suspensions are distinct pharmaceutical dosage forms: a solution requires that the solid particles be dissolved whereas a suspension has solid particles dispersed within it.

184. The specification of the '948 patent also distinguishes between solutions and suspensions. For example, the specification provides examples of both suspensions and solutions. *See* Ex. C, '948 patent at 13:24-27 ("Accordingly, pharmaceutical compositions administered using an MDI typically comprise a solution or suspension of the crystalline freebase active agent in a liquefied propellant."); 14:7-14 ("Suitable pharmaceutical compositions for oral administration may be in the form of capsules, tablets, pills, lozenges, cachets, dragees, powders, granules; or as a solution or a suspension in an aqueous or non-aqueous liquid; or as an oil-in-water or water-in-oil liquid emulsion; or as an elixir or syrup; and the like; each containing a predetermined amount of a crystalline freebase of the invention as an active ingredient."); 15:5-8

("Suitable liquid dosage forms for oral administration include, by way of illustration, pharmaceutically acceptable emulsions, microemulsions, solutions, suspensions, syrups and elixirs.").

185. Additionally, the specification provides processes of preparing a suspension, which makes clear the crystalline freebase is in solid form. *See* Ex. C, '948 patent at 12:51-56 ("Typically, a crystalline freebase active agent is micronized and combined with a suitable carrier to form a suspension of micronized particles of respirable size, where 'micronized particles' or 'micronized form' means at least about 90% of the particles have a diameter of less than about 10 μm."); 13:51-53 ("To prepare a suspension the crystalline freebase active agent is micronized and then combined with the propellant."); 13:59-61 ("Alternatively, a suspension formulation can be prepared by spray drying a coating of surfactant on micronized particles of the active agent"). The specification contains exemplary formulations of a suspension and a solution. The suspension example indicates that the crystalline freebase is micronized to a certain particle size, which indicates the crystalline freebase is in solid form. Ex. C, '948 patent at 18:61-19:14. The solution example, in contrast, indicates the crystalline freebase is dissolved and no longer present in the solution. Ex. C, '948 patent at 19:15-23.

186. The Polymorph Patents family history also illustrates that the applicant knew the difference between solution and suspension. The applicant also obtained suspension claims in U.S. Patent No. 9,226,896, which I understand is in the Polymorph Patents family. Claim 5, for example, is directed to "[t]he composition of claim 1, wherein the crystalline compound is in a suspension." Ex. W, U.S. Patent No. 9,226,896 at claim 5.

187. A POSA cannot understand the claim with reasonable certainty as the claim itself requires an impossibility: a liquid preparation comprising a crystalline solid that can be

characterized by XRPD. Under a POSA's understanding of a solution, there cannot be any solid present. Nor can the claim include a suspension because solutions and suspensions are distinct dosage forms.

188. Turning to Plaintiffs' construction, Plaintiffs have proposed a construction that adds in a process limitation into the claim by including the language "prior to dissolution." However, under Plaintiffs' construction, the claim is still unclear because Plaintiffs' construction still leaves room for a solution to have solid particles. Plaintiffs define solution as "a homogenous mixture of molecules," but in the context of the claim, it is unclear whether Plaintiffs' construction permits the inclusion of undissolved crystalline freebase particles. Thus, even under Plaintiffs' construction, the claim is still not clear to a POSA.

189. Plaintiffs' construction again drops the "regular" component from their "crystalline freebase" construction in their proposed construction of the phrase: "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution." It is unclear what Plaintiffs have dropped the word "regular" in their constructions given that Plaintiffs' construction appears to require that the "crystalline freebase" be in its crystalline state "prior to dissolution."

G. "THE CRYSTALLINE FREEBASE IS IN SOLUTION"

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"the crystalline freebase is in solution" '948 patent, claim 16	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., the freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution is in a homogenous mixture of molecules.	Indefinite To the extent that the Court adopts Plaintiffs' proposed construction, claim 16 is a product-by-process claim

190. I have considered the constructions that are proposed by the parties for "the crystalline freebase is in solution" and in my opinion, based on my experience and the evidence I have considered, a POSA would not be able to understand this claim term with reasonable certainty, which I understand to mean that the claim phrase is indefinite.

191. This phrase includes two claim terms that are also being construed: "crystalline freebase" and "solution." As I discussed above, the proper construction of "crystalline freebase" is "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." The proper construction of "solution" is "liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents."

192. Although a POSA would understand "crystalline freebase" and "solution" terms separately, in the context of the claim itself and as used in the claim phrase, the phrase "the crystalline freebase is in solution" does not make sense to a POSA for the same reasons as discussed above in Section VIII.F for "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution." As illustrated in claim 16 below, the crystalline freebase must be "in solution" and also be characterized by an XRPD pattern.

> 16. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1, and further characterized by having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1, wherein the crystalline freebase is in solution with the pharmaceutically acceptable carrier.

Ex. C, '948 patent at cl. 16 (annotated).

193. As I opined in Section VIII.F, crystalline freebase of revefenacin cannot be "in solution" and be a solid characterized by an XRPD pattern because this is contrary to a POSA's understanding of the term "solution."

194. Turning to Plaintiffs' construction, Plaintiffs have proposed a construction that adds in a process limitation into the claim by including the language "prior to dissolution." However, under Plaintiffs' construction, the claim is still unclear because Plaintiffs' construction still leaves room for a solution to have solid particles. Plaintiffs define solution as "a homogenous mixture of molecules," but in the context of the claim, it is unclear whether Plaintiffs' construction permits the inclusion of undissolved crystalline freebase. Thus, even under Plaintiffs' construction, the claim is still not clear to a POSA.

195. Plaintiffs' construction again drops the "regular" component from their "crystalline freebase" construction in their proposed construction of the phrase: "the crystalline freebase is in solution." It is unclear what Plaintiffs have dropped the word "regular" in their constructions given that Plaintiffs' construction appears to require that the "crystalline freebase" be in its crystalline state "prior to dissolution."

H. "SOLUTION OF A CRYSTALLINE FREEBASE"

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"solution of a crystalline freebase" '948 patent, claim 17	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., a homogenous mixture of molecules of a freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution.	Indefinite To the extent this claim term is not indefinite, "a solution that is prepared by molecularly dispersing a freebase in a solvent such that the freebase is no longer in crystalline form" To the extent that the Court adopts Plaintiffs' proposed construction, claim 17 is a product-by-process claim

196. I have considered the constructions that are proposed by the parties for "solution of a crystalline freebase" and in my opinion, based on my experience and the evidence I have considered, a POSA would not be able to understand this claim term with reasonable certainty, which I understand to mean that the claim phrase is indefinite.

197. This phrase includes two claim terms that are also being construed: "crystalline freebase" and "solution." As I discussed above, the proper construction of "crystalline freebase"

is "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." The proper construction of "solution" is "liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents."

198. Although a POSA would understand "crystalline freebase" and "solution" terms separately, in the context of the claim itself and as used in the claim phrase, the phrase "solution of a crystalline freebase" does not make sense to a POSA for the same reasons as discussed above in Section VIII.F for "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution." As illustrated in claim 17 below, "the solution of a crystalline freebase" requires that the crystalline freebase is characterized by an XRPD pattern or melting point.

> 17. A pharmaceutical composition comprising a solution of a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester characterized by (i) a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1 or (ii) a melting point of about 125° C.

Ex. C, '948 patent at cl. 17 (annotated).

199. As I opined in Section VIII.F, the claims cannot require a solution of a crystalline freebase of revefenacin and also that the crystalline freebase is a solid characterize by an XRPD pattern because this is contrary to a POSA's understanding of the term "solution." Additionally, the claim language creates an additional impossibility because a POSA would not be able to determine the melting point of a solution. A melting point determination necessarily requires the crystalline freebase to be in solid form and not in solution.

200. If a POSA must find some meaning in the claim phrase "solution of a crystalline freebase" the only way to make sense of the phrase is to require the crystalline freebase be in a

solid form before the dissolution process. Thus, a POSA would have to understand the phrase to mean "a solution that is prepared by molecularly dispersing a freebase in a solvent such that the freebase is no longer in crystalline form." A POSA would have to construe "a solution of" to mean that the resulting solution is prepared by dissolving a crystalline freebase. The result of dissolving a crystalline freebase is a solution, and the solution should no longer have any crystalline solid present after the dissolution process occurs.

201. Turning to Plaintiffs' construction, Plaintiffs have proposed a construction that adds in a process limitation into the claim by including the language "prior to dissolution." However, under Plaintiffs' construction, the claim is still unclear because Plaintiffs' construction still leaves room for a solution to have solid particles. Plaintiffs define solution as "a homogenous mixture of molecules," but in the context of the claim, it is unclear whether Plaintiffs' construction permits the inclusion of undissolved crystalline freebase. Thus, even under Plaintiffs' construction, the claim is still not clear to a POSA.

202. Plaintiffs' construction again drops the "regular" component from their "crystalline freebase" construction in their proposed construction of the phrase: "solution of a crystalline freebase." It is unclear what Plaintiffs have dropped the word "regular" in their constructions given that Plaintiffs' construction appears to require that the "crystalline freebase" be in its crystalline state "prior to dissolution."

I. "SOLUTION COMPRISING A CRYSTALLINE FREEBASE"

Disputed Term/Phrase	Plaintiffs' Proposed Constructions	Defendants' Proposed Constructions
"solution comprising a crystalline freebase" '948 patent, claims 18-21	No further construction required. This phrase includes terms already identified for construction above. To the extent that the Court concludes that any further construction is required, this phrase should be construed according to its plain and ordinary meaning to a person of ordinary skill in the art—i.e., a homogenous mixture of molecules comprising a freebase substance having a repeating pattern of molecules that extends over long range in three dimensions prior to dissolution.	Indefinite To the extent that the Court adopts Plaintiffs' proposed construction, claims 18-21 are product-by-process claims

203. I have considered the constructions that are proposed by the parties for "solution comprising a crystalline freebase" and in my opinion, based on my experience and the evidence I have considered, a POSA would not be able to understand this claim term with reasonable certainty, which I understand to mean that the claim phrase is indefinite.

204. This phrase includes two claim terms that are also being construed: "crystalline freebase" and "solution." As I discussed above, the proper construction of "crystalline freebase" is "a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions." The proper construction of "solution" is "liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents."

205. Although a POSA would understand "crystalline freebase" and "solution" terms separately, in the context of the claim itself and as used in the claim phrase, the phrase "solution comprising a crystalline freebase" does not make sense to a POSA for the same reasons as discussed above in Section VIII.F for "crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester in a solution." As illustrated in claims 18 to 21 below, "solution comprising a crystalline freebase" requires that the crystalline freebase is characterized by an XRPD pattern or melting point.

> 18. A pharmaceutical composition comprising a solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a melting point of about 125° C.
>
> 19. A pharmaceutical composition comprising a solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.
>
> 20. A solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a melting point of about 125° C.
>
> 21. A solution comprising a crystalline freebase of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}-ethyl)piperidin-4-yl ester; wherein the crystalline freebase is characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1.

Ex. C, '948 patent at cls. 18-21 (annotated).

206. As I opined in Section VIII.F, a solution cannot comprise a crystalline freebase of revefenacin, where the crystalline freebase is a solid characterized by an XRPD pattern or melting point because this is contrary to a POSA's understanding of the term "solution." The claims are nonsensical because a solution cannot contain solid particles. Additionally, the claim language creates an additional impossibility because a POSA would not be able to determine the melting

point of a solution. A melting point determination necessarily requires the crystalline freebase to be in solid form and not in solution.

207. Turning to Plaintiffs' construction, Plaintiffs have proposed a construction that adds in a process limitation into the claim by including the language "prior to dissolution." However, under Plaintiffs' construction, the claim is still unclear because Plaintiffs' construction still leaves room for a solution to have solid particles. Plaintiffs define solution as "a homogenous mixture of molecules," but in the context of the claim, it is unclear whether Plaintiffs' construction permits the inclusion of particles of undissolved crystalline freebase. Thus, even under Plaintiffs' construction, the claims are still not clear to a POSA.

208. Plaintiffs' construction again drops the "regular" component from their "crystalline freebase" construction in their proposed construction of the phrase: "solution comprising a crystalline freebase" It is unclear what Plaintiffs have dropped the word "regular" in their constructions given that Plaintiffs' construction appears to require that the "crystalline freebase" be in its crystalline state "prior to dissolution."

IX. CONCLUSION

209. For the reasons discussed in my declaration, Defendants' constructions should be adopted because they define the claims in a manner that is consistent with the Polymorph Patents themselves and their respective prosecution histories. Plaintiffs' constructions, in contrast, are overbroad and isolated from the intrinsic record.

210. For the crystalline terms, Defendants' constructions are defined in a way that a POSA would understand "crystalline": a solid freebase with regular repeating pattern of freebase molecules that extends over long-range in three-dimensions. Plaintiffs' omissions of "solid" and "regular" in their definitions of the crystalline terms are incorrect and inconsistent with an understanding of a POSA. There should be no question: crystals are solids and their molecules are

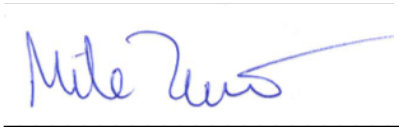
arranged in patterns that are regular. Each of these characteristics are well-accepted and essential components of the definition of a crystal.

211. For the dissolving and solution terms, Defendants' constructions are in line with the use of these terms in the context of the Polymorph Patents, which are directed to pharmaceutical compositions. Thus, a POSA would understand each of these terms in the context pharmaceutical science, rather than a general chemistry understanding (like Plaintiffs present). Defendants' constructions of "dissolving" and "solution" are consistent with the specifications which make clear that the crystalline form must be no longer present, as well as the United States Pharmacopeia. Plaintiffs' constructions are again overbroad and inconsistent with the intrinsic record, since they leave room for partial dissolution. A POSA would expect that crystalline solid will no longer be present after the dissolving process and when in solution. Thus, Defendants' construction of dissolving as "molecularly dispersing a chemical substance in a liquid or mixture of miscible liquids such that the chemical substance is no longer in crystalline form" and solution as "liquid preparation that contains one or more chemical substances dissolved, i.e., molecularly dispersed, in a suitable solvent or a mixture of miscible solvents" should be adopted.

212. With this understanding of the "crystalline" terms, "dissolving", and "solution," a POSA would read the various phrases using these terms in the claims of the '948 patent, and be puzzled. For the claim phrases reciting a crystalline freebase that is dissolved, the specification makes clear that the crystalline solid is no longer present. But the claims themselves require that the crystalline freebase is characterized by XRPD and/or melting point, both of which are indicative of a solid. A dissolved crystalline freebase cannot have a characteristic melting point nor can it be characterized by an XRPD pattern with defined peaks. For the claim phrases that use solution and crystalline freebase, they also are nonsensical. A solution that comprises a crystalline

freebase contradicts the plain understanding of a solution; a solution cannot contain solid particles. A POSA would understand that a pharmaceutical solution must contain dissolved solid; if solid is present, then it is a suspension or a slurry. There is no room for partially dissolved solid, which would be undesirable in a pharmaceutical composition as the amount of solid present would be inconsistent depending upon ambient conditions. Even under Plaintiffs' constructions, there is still lack of clarity because Plaintiffs' constructions leave room for partially dissolved crystalline freebase.

I declare under penalty of perjury that the foregoing is true and correct.



Michael Zaworotko, Ph.D.

Executed this 28th day of February, 2025.

APPENDIX 1

Materials Considered

Exhibit No.	Document	Document Short Name	Bates Nos.
A	U.S. Patent No. 8,541,451	'451 Patent	PLTF-YUP-00000268–289
B (excerpts)	Prosecution History of U.S. Patent No. 8,541,451	Prosecution History of the '451 Patent	PLTF-YUP-00000290–702 (full range)
C	U.S. Patent No. 11,691,948	'948 Patent	PLTF-YUP-00004085–4107
D (excerpts)	Prosecution History of U.S. Patent No. 11,691,948	Prosecution History of the '948 Patent	PLTF-YUP-00004108–4524 (full range)
E	U.S. Patent No. 11,858,898	'898 Patent	PLTF-YUP-00487213–487237
F (excerpts)	Prosecution History of U.S. Patent No. 11,858,898	Prosecution History of the '898 Patent	PLTF-YUP-00487238–487446 (full range)
G	U.S. Patent No. 9,765,028	'028 Patent	PLTF-YUP-00001531–1553
H (excerpts)	Prosecution History of U.S. Patent No. 9,765,028	Prosecution History of the'028 Patent	PLTF-YUP-00001554–2015 (full range)
I	U.S. Patent No. 11,008,289	'289 Patent	PLTF-YUP-00002634–2657
J (excerpts)	Prosecution History of U.S. Patent No. 11,008,289	Prosecution History of the '289 Patent	PLTF-YUP-00002658–2935 (full range)
K	U.S. Patent No. 11,649,209	'209 Patent	PLTF-YUP-00003730–3752
L (excerpts)	Prosecution History of U.S. Patent No. 11,649,209	Prosecution History of the '209 Patent	PLTF-YUP-00003753–4084 (full range)
M	U.S. Patent No. 10,550,081	'081 Patent	PLTF-YUP-00002337–2359
N (excerpts)	Prosecution History of U.S. Patent No. 10,550,081	Prosecution History of the '081 Patent	PLTF-YUP-00002360–2633 (full range)
O	U.S. Patent No. 10,100,013	'013 Patent	PLTF-YUP-00002016–2037

Exhibit No.	Document	Document Short Name	Bates Nos.
P (excerpts)	Prosecution History of U.S. Patent No. 10,100,013	Prosecution History of the '013 Patent	PLTF-YUP-00002038–2336 (full range)
U	Remington's Pharmaceutical Sciences, Chapters 38-39, 50 (2000)	Remington 2000	DEFSREV0001873–1895; DEFSREV0007909–7960
V	U.S. Patent No. 9,415,041	'041 Patent	DEFSREV0007610–7630
W	U.S. Patent No. 9,226,896	'896 Patent	DEFSREV0007589–7609
X	Ralph J. Fessenden & Joan S. Fessenden, Techniques and Experiments for Organic Chemistry 36-49 (1983)	Fessenden 1983	DEFSREV0008070–8088
Y	B. D. Cullity & S. R. Stock, Elements of X-Ray Diffraction (3d ed. 2001)	Cullity 2001	DEFSREV0007763–7823
Z	Byrn et al., Solid State Chemistry of Drugs, 2nd ed. 1999	Byrn 1999	DEFSREV0000678–785
AA	Handbook of Pharmaceutical Analysis (Lena Ohannesian et al. eds., 2002)	Ohannesian 2002	DEFSREV0008117–8190
AB	Allan S. Myerson and Rajiv Ginde, Crystals, Crystal Growth, and Nucleation, Handbook of Industrial Crystallization 33-65 (2d ed. 2002)	Myerson and Ginde 2002	DEFSREV0000007876–7908
AC	Harry G. Brittain, Polymorphism in Pharmaceutical Solids, 1999 (Chapter 6)	Brittain 1999	DEFSREV0000550–621

Exhibit No.	Document	Document Short Name	Bates Nos.
AD	J. Haleblian & W. McCrone, Pharmaceutical Applications of Polymorphism, J. Pharmaceutical Sciences 911 (1969)	Haleblian 1969	DEFSREV0001515–1533
AE	United States Pharmacopeia 31, Chapters 881, 905, 941, 1151, 1160 (2008)	USP 2008	DEFSREV0007966–7988
	Fiese, E.F. & Hagen, T.A., The Theory and Practice of Industrial Pharmacy, Preformulation (Chapter 8) (1986)		DEFSREV0008089–8116
	D. Giron, Ch. Goldbronn, M. Mutz, S. Pfeffer, Ph. Piechon and Ph. Schwab, Journal of Thermal Analysis and Calorimetry, Vol. 68 (2002) 453–465 (Solid State Characterizations of Pharmaceutical Hydrates)		DEFSREV000826–8218
	Madhu Pudipeddi, Abu T.M. Serajuddin, Trends in Solubility of Polymorphs, 929–939 (2005)		DEFSREV0008191–8201
	Hary A. Rose, Erythromycin and Some of Its Derivatives, 938–393 (Vol. 26, No. 5, 1954)		DEFSREV0008202–8203

Exhibit No.	Document	Document Short Name	Bates Nos.
	Vishweshwar, P, et. al., The Predictably Elusive Form II of Aspirin, J. AM. CHEM. SOC, 16802–16803 (Vol. 12, No. 48, 2005)		DEFSREV0008204-8205
	Vogel, A. I., A Textbook of Practical Organic Chemistry, 122–145, Third Edition (1956)		
	Furniss, B.S., et. al., Vogel's Textbook of Practical Organic Chemistry, 1–1514, Fifth Edition (1989)		

APPENDIX 2

Zaworotko CV

Michael John Zaworotko

Contact and www

Mail:	Bernal Chair of Crystal Engineering AD2-021, Dept. of Chemical Sciences and Bernal Institute University of Limerick, Limerick Republic of Ireland
Phone (land):	+353 61 234361
Phone (mobile):	(global): +1 813 857 8322; (Ireland): +353 868144150
E-mail:	xtal@ul.ie
www:	www.ul.ie/crystalengineering
Researcherid:	www.researcherid.com/rid/A-7448-2009
Google Scholar:	scholar.google.com/citations?user=pKuYgikAAAAJ&hl=en&oi=ao
Orcid:	https://orcid.org/0000-0002-1360-540X
Linkedin:	https://www.linkedin.com/in/mike-zaworotko-28497211/
Twitter:	@ZgroupUL

Personal

Date of Birth:	14 August 1956, Tredegar, South Wales
Citizenship:	United Kingdom, Canada, USA and Ireland

Education

1974 - 77	Imperial College, London, U.K., B.Sc. (Hons), ARCS
1978 - 82	University of Alabama, Ph.D. (supervisor: J.L. Atwood)

Professional Experience

1979 - 82	University of Alabama, Research Assistant (Biology Department)
1982 - 85	University of Victoria, Post-Doctoral Fellow (S.R. Stobart)
1985 - 97	Saint Mary's University, Assistant, Associate and Full Professor
1991 - 92	NRC Senior Research Associate (U.S.), USAF Academy
1994 - 97	Saint Mary’s University, Chairperson, Department of Chemistry
1995 - 99	President, Diazans Limited
01/98 - 09/99	Dean of Arts and Science, University of Winnipeg
09/99 - 08/08	University of South Florida, Chair Dept. of Chemistry
08/08 – 10/13	University of South Florida, Professor of Chemistry
11/13-present	University of Limerick, Bernal Chair of Crystal Engineering
06/17-04/22	Co-Director, Synthesis and Solid-State Pharmaceutical Centre

Honors and Awards

Awarded President's Award for Research Excellence, Saint Mary's University, 1994
Appointed Visiting Professor, Universite Louis Pasteur, Strasbourg, 1999
Appointed Visiting Professor, Institute of Biology and Chemistry of Proteins, CNRS, Lyon, France, 2001
Appointed Conference Universitaire de Suisse Occidentale Lecturer, 2002 and 2005
Listed 20th highest citation impact chemist for work published between 2000 and 2010 by Clarivate: http://archive.sciencewatch.com/dr/sci/misc/Top100Chemists2000-10/
Elected Fellow of the American Association for the Advancement of Science, 2011 and appointed in 2012
Appointed Distinguished University Professor, University of South Florida, 2013
Appointed Distinguished Adjunct Professor, King Abdulaziz University, Jeddah, Saudi Arabia, 2014-2015
Listed as highly cited researcher by Clarivate in 2014, 2015, 2016, 2018, 2019, 2022: www.highlycited.com
Institute of Chemistry of Ireland, Eva Philbin Award, 2015
Admitted as Fellow of the Royal Society of Chemistry, 2015
Admitted as Fellow of the Institute of Chemistry of Ireland, 2015
Admitted as Fellow of the Learned Society of Wales, 2017
Appointed as Adjunct Professor at Nankai University, 2017
Science Foundation of Ireland Researcher of the Year Award, 2017
Distinguished Seminar Speaker, City University of Hong Kong, 2018
Admitted as Member of the Royal Irish Academy, 2019
Spiers Memorial Medal, Royal Society of Chemistry, 2021
Ranked the top chemist in Ireland and recognised as a "Chemistry Leader" by Research.com, 2023

Memberships in Professional Organizations

1982 - present	American Chemical Society
2011 - present	American Association for the Advancement of Science
2015 - present	Royal Society for Chemistry
2015 - present	Institute of Chemistry of Ireland
2017 - present	Fellow of the Learned Society of Wales
2019 - present	Royal Irish Academy

Research Programme Overview and Highlights

Zaworotko's research programme focuses upon ***crystal engineering***, the field of chemistry that studies the design, properties and applications of crystals. At the time Zaworotko started his independent career he was one of just a handful of researchers who moved the field forward in the early 1990's through focusing upon the design of new crystals from first principles. Today, crystal engineering addresses properties/applications rather than just design; simply put crystal engineering offers a new paradigm for materials design and discovery. Highlights of Zaworotko's contributions include the following:

1. KEY DISCOVERY IN THE FIELD OF IONIC LIQUIDS

Context: Ionic liquids are salts that exist as liquids below 100°C. The first generation of ionic liquids were of limited utility because they were based upon chlorometallates and therefore tend to chemically decompose in the presence of moisture. **Impact:** The field blossomed rapidly after MZ's 1992 paper (CV paper 60) which introduced **the first examples of air and water stable ionic liquids**. Today, the study of ionic liquids is one of the most impactful areas of chemistry because their low volatility enables a wide range of industrial applications.

2. INTRODUCED PHARMACEUTICAL COCRYSTALS FOR USE IN DRUG PRODUCTS

Context: Crystalline materials lie at the heart of most orally delivered medicines and new crystalline materials can offer intellectual property protection for both new and existing chemical entities. **Impact: The first 2 papers that systematically studied pharmaceutical cocrystals** were published by the Zaworotko group in 2003 (CV papers 177, 181). Today, cocrystals are a topical subject in pharmaceutical science, drug products based upon cocrystals have been introduced by both innovative (four in the past three years) and generic companies (four in the past 2 years). Their relevance has been addressed by a 2018 regulatory guidance from the US FDA.

3. DEVELOPED HYBRID ULTRAMICROPOROUS MATERIALS FOR GAS SEPARATIONS

Context: 40% of industrial energy consumption is currently devoted to commodity purification and storage (e.g. carbon capture, natural gas storage). This represents 15% of the World's energy use and demand for commodities could triple by 2050. Advanced sorbents offer the potential for energy efficient, cost-effective solutions to separations but existing sorbent classes of sorbents are infeasible for most separations and storage applications because of cost, performance and/or stability issues. **Impact: *Hybrid ultramicroporous materials***, HUMs, **were introduced by the Zaworotko group in 2013** (CV paper 276) following on from the discovery of hybrid porous materials in 1995 (CV paper 96). HUMs are so highly selective that they offer 1-2 orders of magnitude better performance vs. existing sorbents such as MOFs and zeolites. HUMs can therefore target difficult challenges in separations such as removal of trace impurities from gas mixtures including direct air capture of CO_2 (CV papers 307, 348) and trace hydrocarbon capture (CV papers 316, 377, 419).

4. TECHNOLOGY TRANSFER

Context: Creation of intellectual property and transferring it can enable companies to make the investments necessary to develop new technologies and products. **Patents:** 25 patents have issued, 9 of which have been licensed. In addition, multiple patents are pending since moving to Ireland in Nov 2013. Of note, our novel anhydrate crystal form of EGCg was studied as a treatment against ulcerative colitis, a lithium cocrystal in in Phase II clinical trials (depression) and our patents on carbon capture are attracting the attention of the petroleum industry. **Advisory role:** MZ has served on a number of small company scientific advisory boards and he has consulted with most of the large pharmaceutical companies.

Research Grants Awarded

1986 "Organotransition Metal Sustained Liquid Clathrates." NSERC Operating - $16,320 - one year.
"Liquid Clathrates - Utilization as Alkylating Agents." NSERC Research Development - $12,000/yr. - two years.
"Metal Superoxide Complexes" NSERC General - $4,000 - one year
"Homogeneous Catalysis using Liquid Clathrates." Senate Research - $1,500 and $1,450 - 1 year.
SEED Summer Employment Subsidy - ca. $2,800

1987 "Organotransition Metal Sustained Liquid Clathrates." NSERC Operating - $16,320 - one year.
"Determination of the Alkylating Capabilities of Anionic Alkyl Aluminum Reagents."
Petroleum Research Fund (type B) - U.S. $10,000/yr. - two years.
"Room Temperature Ionic Liquids." NSERC General - $2,500 - one year.
"Novel Applications of Liquid Clathrates." Senate Research - $1,756 - one year.
SEED Summer Employment Subsidy - ca. $1,500.

1988 "Salt Sustained Liquid-Liquid Binary Phases." NSERC Operating - $10,000 - one year.
"Novel Approaches to Hydrocarbon Separation Problems." Imperial Oil Ltd. - $10,000 - 1 year.
"Applications of Room Temperature Liquids." NSERC General - $4,000 - one year.
"Applications of Ionic Liquids to Separation Problems." Senate Research - $1,600 - one year.
SEED Summer Employment Subsidy - ca. $2,000

1989 "Aspects of Arene Chemistry." NSERC Operating - $25,844 per year for 3 years.
"Hydrocarbon Separation Properties of Molten Salts." Imperial Oil Ltd. (URG) - $8,000 - 1 year.
"Novel Approaches to Lubricating Oil Processing." NSERC University/Industry with Imperial Oil - $50,000 per year for 3 years.

1990 An internal grant of $116,000 was awarded for purchase of a single crystal X-ray diffractometer.

1991 "X-ray crystallographic work station." NSERC Equipment - $25,193

1992 "Arenes: Covalent and Non-covalent Bonding." NSERC operating - $26,810 per year for four years.
"Ionic Liquids" NSERC General - $2,500

1993 "Crystal Engineering of Diamondoid Networks with Zeolite-Like Physical Features."
NSERC University/Industry with ICST - $60,000 - 1 year.

1994 "Crystal Engineering of Microporous Polymeric Solids." ICST and NSERC - $89,000/year - 2 years.

1995 "Novel Biologically Active Compounds." Contract from Shaw Group Ltd. - $100,000 - 1 year

1995 "Crystal Engineering: The Design and Application of Functional Solids."
NATO ASI - $80,000 (co-chair with K. Seddon)

1996 "Crystal Engineering of Functional Solids." NSERC - $40,740 per year - 4 years

1996 "Low temperature device for single crystal X-ray diffractometer." NSERC Equipment - $33,730.

1996 "Novel Biologically Active Compounds." Contract from Shaw Group Ltd. and IRAP - $150,000 - 1 yr

1996 "Guest Interactions in Crystal Engineered Host Frameworks." NATO CRG - $11,300 (with K. Seddon, R. Rogers)

1996 "Environmental Applications of Organic Clays." ESTAC and NSERC - $38,300 - 1 year

1996 "CCD diffractometer." Internal Research Grant - $240,000

1998 "Novel Technology for Hog Manure Remediation." – $35,000 – 1 year
Manitoba Livestock Manure Management Initiative

1998 "Environmental Applications of Organic Clays." – $75,000 – 1 year
ESTAC/NSERC (with M. Lamoureux, Saint Mary's university)

Research Grants Awarded -- Continued

2000 "Biocomposite Materials by Design." – $20,000 – 1 year – Florida High Technology Corridor (with D. Merkler, University of South Florida)

2000 "Characterization of Polymorphs of Fluocinolone Acetonide." - $20,000 – 1 year Bausch & Lomb Pharmaceuticals, Ltd., Tampa.

2001 "From Molecular Polygons to Discrete Faceted Polyhedra to Porous Frameworks." – $445,219 – 04/15/01 thru 03/31/04 – National Science Foundation (Division of Materials 0101641).

2001 "Synthesis, X-ray Study and Inclusion Properties of the Crown Based Extended Networks." - $35,000 – 01/01/02 thru 04/30/03 – US Civilian Research and Development Foundation (with Y. Simonov, Moldova, 20% of funds to M.Z.).

2001 "Intelligent Design of Pharmaceutical Solids." - $40,000 – 06/01/02 thru 05/31/03 – Vahlteich Research Fund (with N. Rodriguez, University of Michigan, 40% of funds to M.Z.).

2002 "Multi-Component Crystalline Pharmaceutical Phases." - $35,000 – 01/01/03 thru 12/31/03– Transform Pharmaceuticals

2003 "Integrated Interdisciplinary Nanoscience REU." – $213,000 – 01/01/03-12/31/05 – National Science Foundation (PI with co-PI's R. Walsh and P. Muisener and 7 faculty mentors.

2003 "Molecular Nanoscience Research at USF – NanomolUSF." - $50,000 – 05/01/03-04/30/05 – Interdisciplinary Research Grant Program at USF (PI with co-PI's S. Hariharan and R. Schlaf)

2003 "Multi-Component Crystalline Pharmaceutical Phases." - $320,000 – 08/26/03 thru 08/25/07– Transform Pharmaceuticals.

2003 "Novel Agricultural Chemical Formulations." - $12,000 – 09/01/03 thru 08/31/04 – Florida High Tech Corridor.

2004 "Smart Porous Metal-Organic Frameworks (MOFs) for Hydrogen Recovery and Storage." - $200,000 – 01/01/05-12/31/05 – NASA (M. Eddaoudi PI, co-PI with B. Space, J. Eckert, A. Raissi, N. Mohajeri).

2005 "Purchase of a High Resolution ESI-TOF Mass Spectrometer."- $273,754 – 02/01/05-01/31/06 – NSF-CRIF (PI and major user with six other chemistry faculty).

2006 "Smart Porous Metal-Organic Frameworks (MOFs) for Hydrogen Recovery and Storage." - $125,000 – 01/01/06-12/31/06 – NASA (M. Eddaoudi PI, co-PI with B. Space, J. Eckert, A. Raissi, N. Mohajeri).

2006 "Design of pharmaceutical co-crystals." - $53,300 – 02/01/06-08/31/07 – Transform Pharmaceuticals.

2007 "Novel Porous Metal Organic Frameworks (MOF) for Hydrogen Storage." - $882,000 – 08/15/07-08/14/10 – Department of Energy (M. Eddaoudi PI, co-PI with B. Space, J. Eckert, A. Raissi)

2008 "Functional Photocatalytic Materials for Threat Decontamination/Degradation." - $2,400,000 – 07/10/08-07/09/12 – Department of Defense (R. Larsen PI, M. Zaworotko is co-PI with M. Eddaoudi, J. Harmon and P. Zhang) .

2008 "Pharmaceutical Co-Crystals for Alzheimer's Disease Therapy: Improving Clinical Properties Through Crystal Engineering." – $59,786 –07/01/08-06/30/09 - Byrd Alzheimer's Institute (M. Zaworotko PI, T. Gauthier co-PI).

Research Grants Awarded -- Continued

2009 "Hydrogen Storage Materials with Binding Intermediate between Physisorption and Chemisorption." - $55,000- 08/17/09-08/16/10 – Department of Energy (PI of sub-contract from University of California, Santa Barbara).

2010 "KAUST-USF Metal-Organic Materials Network: Smart Materials for Energy and Environmental Sustainability." – $1,003,505 – 09/20/10-09/19/13 – KAUST Faculty Initiated Collaboration – (Zaworotko is PI with co-PI's B. Space, P. Zhang and J. Eckert).

2011 "Design, synthesis and properties of calixarene based metal organic materials, calix-MOMs." – $88,000 - 11/01/11-10/31/13 - CRDF Global CGP 2010/2011 Climate Change & Energy (with Institute of Organic Chemistry, National Academy of Sciences of Ukraine, Zaworotko is USF PI).

2011 "Electric Powered Adsorption Heat Pump for Electric Vehicles." - $200,000 – 12/01/11-11/30/2013 - Department of Energy ARPA-E HEATS (with Pacific Northwest Laboratory (Zaworotko is USF PI with co-PI Shengqian Ma).

2013 "Crystal Engineering of Task-Specific Materials." - €5,000,000 - 11/01/13 - 10/31/18 - Science Foundation of Ireland Research Professor Programme

2016 "Solid-State Ionic Cocrystal Complexes." - €40,000 – 06/01/16 - 08/31/16 – Syngenta.

2016 "Advanced Sensor Development." - €35,000 – 09/01/16 - 08/31/17 – Analog Devices.

2016 "Solid-State Ionic Cocrystal Complexes." - €50,000 – 12/08/16 - 12/07/17 – Syngenta.

2017 "Solid-State Ionic Cocrystal Complexes." - €50,000 – 12/12/17 - 12/11/18 – Syngenta.

2018 "Trace CO_2 Removal for Natural Gas Liquefaction by Advanced Physisorbent Materials." – $172,520 out of $400,000 total budget – 05/01/18-04/30/21 – Qatar National Research Foundation.

2018 "GRACE - Green Adsorbents for Clean Energy." - €1,901,505 – 01/01/18 – 12/31/22 – Science Foundation of Ireland Investigators Programme.

2018 "AQUASORB - Advanced Sorbents for Water Capture, Storage and Release" – €179,000 – 09/01/18-08/31/19 – Molecule RnD, Industry funded research project.

2019 "MAGSORB - Magnetically Switching Physisorbents for Water Capture" - €179,000 - 04/01/19-03/31/20 – Molecule RnD, Industry funded research project.

2019 "SSPC-PharM^5" – €23,129,949 – 06/01/19-05/31/2025 – Science Foundation of Ireland Research Centres Phase 2 (Zaworotko is PI with eleven co-PIs).

2019 "SALMA – Switching Adsorbent Layered Materials" - €961,191 – 09/01/19-08/31/2023 – Irish Research Council Laureate Award.

2019 "AQUASORB-II - Advanced Sorbents for Water Capture, Storage and Release" – €200,000 – 09/01/19-08/31/20 – Molecule RnD, industry funded research project.

2020 "C-MINUS – Low energy carbon capture devices to enable on-site carbon capture and net negative carbon technologies" - €220,000 – 01/01/20-12/31/20 – SFI Zero Emissions Challenge.

2020 "LIGHTSORB – Light-induced Switching Physisorbents" - €157,794 – 02/01/20-01/31/20 – Molecule RnD, industry funded research project.

2020 "SYNSORB – SYNergistic SORBents" – €2,497,298 – 09/01/20 – 08/31/25 – ERC Advanced.

2020 "AQUASORB-III" – €528,000 - 10/01/20-09/30/21 - Molecule RnD, industry funded project.

2022 "AQUASORB-IV" – €191,000 - 02/01/22-01/31/23 - Molecule RnD, industry funded project.

2023 "AQUASORB" - €364,127 – 05/01/23-04/30/26 – SFI-NSF Tripartite.

2024 "DACSORB" - €55,000 – 11/01/23-04/30/24- Aspiradac, industry funded project.

Invited Seminars to Companies and Universities

1987	. Mount Allison University
	. CANMET, Ottawa
	. **Imperial Oil Limited, Sarnia**
	. University of Waterloo
	. Dalhousie University
1988	. Acadia University
	. University of South Alabama
	. University of Alabama
1989	. Northern Illinois University
	. Brown University
1990	. University of Saskatchewan
	. Dalhousie University
1992	. **Imperial Oil Limited, Sarnia**
	. CANMET, Ottawa
	. University of Alabama
	. University of Mississippi
	. Wright Laboratory, Dayton, Ohio
	. Memorial University of Newfoundland*
1993	. Dalhousie University*
	. University of New Brunswick*
	. Universite Moncton*
	. University of Prince Edward Island*
	. Mount Allison University*
	. University of Guelph
	. University of Groningen
	. University of Alabama
1994	. Acadia University
	. University of Northern British Columbia
	. **Exxon Research and Engineering, N.J.**
1995	. ETH Zurich
	. The Queen's University of Belfast
	. The University of Birmingham
	. The University of Western Ontario
	. The University of Windsor
	. Brown University
1996	. Brown University
	. Northern Illinois University
	. National Research Council, Ottawa
	. Trinity College, Dublin, Ireland
1997	. The University of Waterloo
	. CNRS, Lyon, France

Invited Seminars -- Continued

1997
. University of Siegen, Germany
. Saint Mary's University

1998
. The University of Winnipeg
. The University of Manitoba
. Sumi State University
. Institute of Physics of Ukraine
. Kyiv State University
. **Trojan Technologies, London, Ontario**
. **Nortran Pharmaceuticals, Vancouver**
. **Apotex, Winnipeg**
. The University of Missouri, Columbia
. The University of Manitoba (Physics)

1999
. Seoul National University, Korea
. POSTECH, Pohang, Korea
. University of South Florida
. Universite Louis Pasteur, Strasbourg, France
. **Eastman Chemical, US**
. **Bell Labs**
. University of Windsor, Canada
. **Constellation Technologies, Florida**

2000
. Memorial University of Newfoundland, Canada
. Acadia University, Canada
. Mount Allison University, Canada
. Saint Mary's University, Canada
. University of Miami
. University of Florida
. Clemson University
. University of Alabama
. University of Mississippi
. University of South Carolina
. University of Prince Edward Island, Canada
. Saint Francis Xavier University, Canada

2001
. University of Michigan
. Universite Claude Bernard, Lyon, France
. University of Winnipeg, Canada

2002
. University of South Florida, College of Medicine
. University of Iowa
. University of North Carolina
. Worcester Polytechnic Institute
. **Transform Pharmaceuticals**
. University of Durham, England
. University of Geneva, Switzerland

Invited Seminars -- Continued

2002	. University of Neuchatel, Switzerland
	. University of Fribourg, Switzerland
	. University of Bern, Switzerland
	. University of Winnipeg, Canada
	. University of Brandon, Canada
2003	. **World Precision Instruments, Sarasota**
	. University of South Florida, Complex Systems Seminar Series
	. University of Manitoba
	. University of Prince Edward Island
	. Mount Allison University
	. Saint Mary's University
2004	. Georgetown University
	. Institute for Chemical and Engineering Sciences, Singapore
	. **Pfizer Global Research, Groton, CT**
	. Institut de Biologie et Chimie des Proteines, Lyon, France
	. Acadia University
	. St. Mary's University
	. **Amgen, Thousand Oaks, California**
2005	. Davidson College, NC
	. University of Houston
	. **Amgen, Cambridge, Massachusetts**
	. **Johnson & Johnson Pharma R&D, Raritan, NJ**
	. University of Notre Dame
	. University of Basel
	. University of Geneva
	. University of Bern
	. University of Lausanne
	. University of Notre Dame
2006	. University of Texas, San Antonio
	. University of Cape Town
	. University of Stellenbosch. S. Africa
	. **AstraZeneca, Macclesfield, UK**
	. **Novartis, E. Hanover, New Jersey**
2007	. Florida International University
2008	. Acadia University, Canada
2009	. Saint Mary's University, Canada
	. Sun Yat-Sen University, China
	. National Taiwan University, Taipei, Taiwan
	. Academia Sinica, Taiwan
	. National Chung Cheng University, Taiwan
2010	. National University of Morelos, Cuernvaca, Mexico
	. Northwestern University

Invited Seminars -- Continued

2010	. Tulane University Medical School, Peptide Research Group
	. **Mutual Pharma, Philadelphia**
	. **Cephalon, Frazer, PA**
	. College of New Jersey, Ewing, NJ
	. Nanjing University, China
	. Southeast University, China
2011	. National Chemical Laboratory, Pune, India
	. **Dr. Reddy's, Hyderabad, India**
	. University of Hyderabad, India
	. Nankai University, China
	. Texas A&M University
	. University of Michigan
	. University of West Florida
2010	. Northwest University, X'ian, China
	. Luoyang Normal University, Luoyang, China
2011	. Indian Institute of Technology, Kharagpur, India
	. Indian Association for the Cultivation of Science, Kolkata, India
	. Indian Institute of Science Education and Research, Kolkata, India
	. Technical University of Delft, The Netherlands
	. **Solvias AG, Basel, Switzerland**
2012	. University of Milan Bicocca, Italy
	. **Alkermes, Inc., Waltham, MA, USA**
	. National University of Singapore
	. Institute of Chemical and Engineering Sciences, Singapore
	. Saint Francis Xavier University, Canada
	. Saint Mary's University, Canada
	. East China Normal University, Shanghai
2013	. Pacific Northwest National Laboratory
	. University of Malaya
	. Tianjin Normal University
	. Tianjin University of Technology
	. Nankai University
	. Bohai University
2014	. University College Cork, Ireland
	. Jiangnan University, China
	. Nanyang Technological University, Singapore
	. **Alkermes, Inc., USA**
	. Cinvestav, Mexico City
	. University of Windsor, Canada
	. University of Nottingham, UK
	. Queen's University, Belfast, UK

Invited Seminars -- Continued

2015	. King Abdulaziz University, Saudi Arabia
	. Herriot Watt University, Scotland
	. Cardiff University, Wales
	. Universidade Federal do Ceara, Fortaleza, Brasil
	. Universidade Sao Paolo, Sao Carlos, Brasil
	. McGill University, Canada
	. **Moderna Therapeutics, Boston, USA**
	. Saint Mary's University, Canada
2016	. National University of Ireland, Galway
	. **Syngenta, Jealott's Hill, UK**
	. Nankai University (New campus)
	. Shanghai Jiao Tong University
	. Nankai University (Old campus)
	. National Chemical Laboratory, Pune, India
	. IISER Pune, India
	. University of Jyväskylä, Finland
	. Dalian University of Technology, China
	. **F. Hoffmann-La Roche, Basel, Switzerland**
2017	. University of Leeds
	. Tianjin University of Technology
	. Nankai University (College of Chemistry)
	. Nankai University (College of Materials Science and Engineering)
	. Zewail City of Science and Technology, Center for Materials Science
2018	. **Zentiva, Prague**
	. Tianjin University of Technology
	. Nankai University (College of Chemistry, twice)
	. Nankai University (College of Materials Science and Engineering)
	. Northwest Polytechnical University, Xi'an
	. Shanxi Normal University
	. Trinity College, Dublin
	. **Pharmaron, Beijing**
	. City University of Hong Kong (Distinguished Seminar Speaker)
2019	. **Pfizer, Groton, CT**
	. **Vertex Pharmaceuticals, Boston**
	. **Alkermes, Waltham**
	. Nankai University, College of Chemistry.
	. Guangxi Normal University
	. Tianjin University
	. Cambridge University
	. University of Fribourg
	. Nankai University College of Pharmacy
	. **Syngenta, Jealotts Hill, UK**

Invited Seminars -- Continued

2022	. University of North Texas
2023	. Beijing University of Science and Technology
	. Yunnan Normal University
	. Nankai University Chemistry
	. Nankai University Materials Science and Engineering
	. Tianjin University
2024	. University Mohamed 6 Polytechnic, Morocco
	. Lanzhou University
	. Peking University
	. Chinese Academy of Sciences, Beijing
	. Nankai University Chemistry
	. Nankai University Materials Science and Engineering
	. China University of Petroleum, Beijing

Synergistic Activities

- Industrial interaction since 1998. Funded research with the following companies: Bausch and Lomb Pharma; Constellation Tech.; Breed Technologies; Transform Pharma; United Agricultural Services, Analog Devices, Zentiva; Syngenta; Jazz Pharma; Molecule R&D; Aspiradac.
- Served as scientific consultant for several innovative pharmaceutical companies.
- Served as legal consultant in >30 patent litigation cases since 2006.
- Organizer of 17 symposia or conferences in U.S./Canada.
- Member of UTEK Corporation's Scientific Advisory Council; 2003-2007 - Member of Transform Pharmaceutical's Scientific Advisory Board; 2007-2010 - Member of Thar Pharmaceuticals Scientific Advisory Board; 2012 – 2016 - Member of Alkermes, Inc., Scientific Advisory Board; 2016-present, Member of MOF Technologies Scientific Advisory Board.
- Founding Editor of *Crystal Engineering*, published by Elsevier from 1997-2003; Editorial Board *J. Chemical Crystallography*, *Crystal Growth & Design, International Union of Crystallography Journal*; International Advisory Committee: VIII International Seminar on Inclusion Compounds 2002, Singapore Int. Chemistry Conference 2005.
- Associate Editor of *Crystal Growth & Design*, published by the ACS, July 2006-Dec 2018.
- Papers regularly reviewed for many journals including *Science*, *Nature*, *JACS*, *Angewandte Chemie*.
- Served on panels for Canadian Foundation for Innovation and NSF/EPSCOR in 2003, MRSEC preproposal committee for NSF (2004), Canadian Foundation for Innovation in 2006 and 2009, Canada Research Chairs Program (2008) AAAS Materials for Energy Applications Panel (2010), NSF CAREER Awards Panel (2010), External Advisory Board for NYU-Xavier PREM (2011), Science Foundation of Ireland (2012), NSERC Canada Grant Selection Panel (2012-2015), NSF DMR "MOF panel" (2013), ERC Advanced Grant Panel (2016, 2018, 2020, 2022), ERC SAP panel for PE5 (2022).
- External Reviewer for the Following Theses: M.S. (Memorial University, 1990; University of Cape Town, 1996). Ph.D. (Saskatchewan, Canada, 1990; Alabama, 1993, Waterloo, Canada, 1997; Chinese University of Hong Kong, 1999; University of Windsor, Canada, 1999; University of Hyderabad, 2003; University of Pune, India, 2008; IIT Guwahati, India, 2009; Technical University of Delft, Netherlands, 2011; University of Windsor, Canada, 2014; USP Sao Carlos, Brasil, 2015; University of Milan, 2016; University of Fribourg, 2019; National University of Singapore, 2019; Manchester, 2020; University of Cape Town, 2020, American University of Beirut, 2021).
- Served as external reviewer of the chemistry program at Lebanese American University (2009).

Zaworotko Group Members (February 2025)

Five post-graduate (PhD) students, one as sole supervisor (Samuel Shabangu), four as joint supervisor (Hooman Hasheminejad, Asif Raza, Bhawna Kumari, Qihao Yin);
Six post-doctoral fellows (Tao He, Alan Eaby, Lilia Crotor, Bharti Singh, Ying Liu, Aizhamal Subanbekova).

Leadership Experience

09/94-12/97: Chair, Department of Chemistry, Saint Mary's University.
Highlights: Academic leadership of Department at a time of faculty renewal and growth in majors and external funding.
01/98-09/99: Dean of Arts and Science, University of Winnipeg.
Highlights: Administrative leadership for 90% of faculty and programs at University of Winnipeg, responsible for strategic resource allocation within College of Arts and Science.
09/99-08/08: Chair, Department of Chemistry, University of South Florida.
Highlights: Accountable officer for a Department in one of the largest universities in the U.S. (>47,000 students at the time). Department graduated ca. 300 majors per year, served >130 graduate students, had 27 tenure track faculty and 16 permanent staff during my tenure. Developed a 5-year plan that resulted in the hire of 11 new faculty between 2000 and 2009. Departmental research grants increased to ca. $5,000K/year (up from ca. $400K/year in 1999). Responsible for fund raising and external relations.
06/17-04/22: Co-Director of the Synthesis and Solid-State Pharmaceutical Centre (SSPC).
Highlights: Provision of scientific leadership to a global hub of pharmaceutical and process innovation with 24 industry partners, 9 research performing organisations and 8 full-time employees. SSPC was renewed for six years starting June 2019. Cash budget for operations and basic research is >€6 million/year.

Evidence of Impact

- Listed **20th highest citation impact chemist** for work published between 2000 and 2010 by Thomson Reuters on 02/10/11.
- Listed as **highly cited researcher** in Chemistry (out of *ca*. 200 worldwide) by Thomson Reuters in 2014, 2015 and 2016: www.highlycited.com Listed by Clarivate Analytics in 2018 in two fields, "chemistry" and "pharmacology/toxicology", in 2019 and 2022 in "cross-field" category.
- **Invited lectures** (including multiple plenary and keynote lectures) at 115+ regional, national or international meetings from 2012 thru the present.
- **Invited seminars** at 70+ universities (US, Canada, UK, France, Switzerland, China, Ireland, Taiwan, India, The Netherlands) and 11 companies from 2012 thru the present.
- **H-index of 120**, over 65200 citations including >19000 since January 2020 (*Google Scholar*).
- A 2001 review article was determined by ISI to be the **#1 hot paper in Chemistry** in July 2003 and in October 2007 was recognized by ACS as a highly cited paper: http://pubs.acs.org/journals/chreay/promo/most/highly_cited/2007/oct.html
- Featured in a *C&E News* articles on metal-organic materials (09/15/03), polymorphism in drugs (11/21/05) and pharmaceutical co-crystals (18/07/07).
- Nine patents licensed including three with 6 figure license fees.
- All but one of my former graduate students are professionally employed in industry or academia.
- Three of my former students are now faculty members who were awarded NSF CAREER Awards (Dr. **Len MacGillivray**, Iowa; Dr. **K. Travis Holman**, Georgetown, Dr. **Z. Wang**, South Dakota); Thirteen more of my former graduate students or post-doctoral fellows are now tenured or tenure-track faculty members: **G. McManus**, Florida Gulf Coast; **H. Abourahma**, College of New Jersey; **Z. Zhang**, Nankai University; **A. Schoedel**, Florida Institute of Technology; **K.J. Chen**, Northwestern Polytechnical University; **Q. Yang**, Xi'an Jiaotong University; **Zhuxiu Zhang**, Nanjing Tech University); **S.Y. Zhang** (Zhejiang University); **S. Chen** (Lanzhou University); **Y. Peng** (China University of Petroleum); **S. Elsaidi** (Illinois Institute of Technology); **M. Mohamed** (Illinois Institute of Technology); **S. Mukherjee** (University of Limerick).
- Highlights. (a) **PhD students: Z. Zhang** won an *ACS DIC Young Investigator Award* in 2015 and a *1000 Young Talent Award* in 2016; **A. Schoedel** was one of 45 finalists for the Reaxys Ph.D. Prize in 2014; **M. Shivanna** was awarded a JSPS post-doctoral fellowship in 2019; **K. Koupepidou** was awarded a JSPS post-doctoral fellowship in 2025. (b) **Post-doctoral Fellows:** In 2017, **A. Bajpai**, was awarded an *Inspire Fellowship* from DST, India (19 awards from > 1000 applicants); In 2016, **M. Lusi**, won a SIRG Fellowship from Science Foundation Ireland to support independent research; **K.J. Chen** was awarded a *1000 Young Talent Award* in 2017; **Q. Yang** was awarded a *Young Talent Award* in 2018; **N. Sikdar** and **S. Mukherjee** were awarded Alexander von Humboldt Fellowships in 2018 and 2019, respectively; **C. Hua** was selected *2018 McKenzie Postdoctoral Fellow* at the University of Melbourne and a *CAS Future Leader* in 2020; **A. Bezrukov** was awarded a *Research Ireland Industrial Fellowship* in 2024.
- **Cover art**: *Crystal Growth & Design* (2004, 2015), *ChemComm* (2001, 2004, 2015 x 2, 2017, 2020, 2023), *Chemical Reviews* (2001), *Chemistry: A European Journal* (2020), *Chemical Society Reviews* (2009).
- > $40 million in external funding (either as PI or co-PI) since 2000.

PUBLICATIONS

Peer Reviewed Papers

C = Communication; F = Full Paper; N = Note; L = Letter; Corresponding author underlined.

1. (C) Guzman, C.E.; Wilkinson, G.; Atwood, J.L.; Rogers, R.D.; Hunter, W.E.; Zaworotko, M.J. "Synthesis and Crystal Structures of Chloro(trimethylphosphine)tris(trimethylsilymethyl) molybdenum(IV) and Di-μ-chloro-bis[bis(carbonyl)tri-methylphosphine(1-2-η-trimethylsilymethyl-carbonyl)molybdenum (II)]." *J. Chem. Soc., Chem. Comm*., 465, 1978.

2. (F) Shakir, R.; Zaworotko, M.J.; Atwood, J.L. "The Crystal and Molecular Structure of $K[Al_2(CH_3)_6SCN]$, a Compound which Contains an S,N-bridging Thiocyanate Ligand." J*. Organomet. Chem*., 171, 9, 1979.

3. (F) Shakir, R.; Zaworotko, M.J.; Atwood, J.L. "Crystal and Molecular Structure of Cesium Isothiocyanatotrimethylaluminate." J*. Cryst. Mol. Struct*., 9, 135, 1979.

4. (F) Zaworotko, M.J.; Atwood, J.L.; Floch, L. "Crystal and Molecular Structure of 5-amino-1,2,3,4,-thiatriazole." *J. Cryst. Mol. Struct.,* 9, 2173,1979.

5. (F) Zaworotko, M.J.; Atwood, J.L. "Crystal and Molecular Structure of $Cl_2AlN(C_2H_4N(CH_3)_2$." *Inorg. Chem.,* 19, 268, 1980.

6. (C) Cetinkaya, B.; Hitchcock, P.B.; Lappert, M.F.; Torroni, S.; Atwood, J.L.; Hunter, W.E.; Zaworotko, M.J. "Transition-metal Complexes of Two Valence Tautomers of a Bulky Phenoxide, 2,6-Bu-t_2-4-MeC_6H_2O- (ArO-); Preparation and Crystal and Molecular Structure of a Phenoxytitanium (III) and a Cyclohexadienonyl Rhodium(I) Complex, [Ti($\eta C_5H_50_2$)OAr] and [Rh(ArO ^{5})($PPh_3)_2$]." *J. Organomet. Chem.,* 188. C31, 1980.

7. (F) Rausch, M.D.; Hart, W.P.; Atwood, J.L.; Zaworotko, M.J.; "The Formation and Molecular Structure of (η^5-nitrocyclopentadienyl) dicarbonyl rhodium." *J. Organomet. Chem.,* 197, 225, 1980.

8. (F) Guzman, E.C.; Wilkinson, G.; Rogers, R.D.; Hunter, W.E.; Zaworotko, M.J.; Atwood, J.L. "Synthesis and Crystal Structures of Chloro (trimethylphosphine) tris(trimethylphosphine)(1-2-trimethyl-silylmethyl-carbonyl)molybdenum (II)." *J. Chem. Soc. Dalton Trans.,* 229, 1980.

9. (C) Lappert, M.F.; Slade, M.J.; Atwood, J.L.; Zaworotko, M.J. "Monomeric Coloured Germanium (II) and Tin (II) Di-t-butylamides, and the Crystal and Molecular Structure of $Ge(NCMe_2[CH_2]_3CMe_2)_2$." *J. Chem. Soc., Chem. Comm.,* 621, 1980.

Peer Reviewed Papers -- Continued

10. (C) Cetinkaya, B; Gumrukcu, I; Lappert, M.F.; Atwood, J.L.; Rogers, R.D.; Zaworotko, M.J. "Bivalent Germanium, Tin and Lead 2,6-di-tert-butylphenoxides and the Crystal and Molecular Structures of $M(OC_6H_2Me\text{-}4\text{-}But_2\text{-}2,6)_2$ (M = Ge or Sn)." *J. Amer. Chem. Soc.,* 102, 2088, 1980.

11. (F) Stobart, S.R.; Dixon, K.D.; Eadie, D.T.; Atwood, J.L.; Zaworotko, M.J. "Transition Metal Complexes and Pyrazolyl Bridging Ligands between Very Different Metal Centers." *Angew. Chem. Int. Ed. Eng.,* 19, 931, 1980.

12. (C) Lappert, M.F.; Miles, S.J.; Atwood, J.L.; Zaworotko, M.J.; Carty, A.J. "Oxidative Addition of an Alcohol to the Alkylgermanium(II) Compound $Ge[CH(SiMe_3)_2]_2$; Molecular Structure of $Ge[CH(SiMe_3)_2]_2(H)OEt$." *J. Organomet. Chem.,* 212, 1981, C4.

13. (F) Lappert, M.F.;, Riley, P.I.; Yarrow, P.I.W.; Atwood, J.L.; Hunter, W.E.; Zaworotko, M.J. "Metallocene Derivatives of Early Transition Metal Elements. Part 3. Synthesis, Characterization, Conformation and Rotational Barriers ($Zr\text{-}C_{sp}^3$) of the Zirconium (IV) Chlorides [$Zr(\eta\text{-}C_5H_4R)_2CH(SiMe_3)_2$)C1] and the Crystal and Molecular Structures of the t-butyl and Trimethylsilyl complexes (R = Me_3C or Me_3Si)." *J. Chem. Soc. Dalton Trans.,* 314, 1981.

14. (C) Beveridge, K.A.; Bushnell, G.W.; Dixon, K.R.; Eadie, D.T.; Stobart, S.R.; Zaworotko, M.J.; Atwood, J.L. "Pyrazolyl-bridged Iridium Dimers. 1. Accommodation of Both Weak and Strong Metal-metal Interactions by a Bridging Pyrazolyl Framework in Dissymmetric Dimeric Structures." *J. Amer. Chem. Soc.*, 104, 920, 1982.

15. (C) Coleman, A.W.; Eadie, D.T.; Stobart, S.R., Atwood, J.L.; Zaworotko, M.J. "Pyrazolyl-bridged Iridium Dimers. 2. Contrasting Modes of Two Center Oxidative Addition to a Bimetallic System and Reductive Access to the Starting Complex: Three Key Diiridium Structures Representing Short Non-bonding and Long and Short Bonding Metal-metal Interactions." *J. Amer. Chem. Soc.,* 104, 922, 1982.

16. (F) Zaworotko, M.J.; Shakir, R.; Atwood, J.L.; Sriyunyongwat, V.; Reynolds, D.S.; Albright, T.A. "Synthesis and Structure of Dicarbonyl(η^5-methylcyclopentadienyl)triphenylphosphine manganese(I)." *Acta. Cryst.,* B38, 1572, 1982.

17. (F) Zaworotko, M.J.; Rogers, R.D.; Atwood, J.L. "Interaction of Trimethylaluminum and Trimethylgallium with the Acetate Anion. Synthesis and Crystal Structures of $[N(CH_3)_4][Al_2(CH_3)_6CH_3COO]$ and $Rb[Ga_2(CH_3)_6CH_3COO]$." *Organometallics,* 1, 1179, 1982.

18. (F) Eadie, D.T.; Dixon, K.R.; Stobart, S.R.; Zaworotko, M.J.; Atwood, J.L. "Crystal and Molecular Structures of Tetrafluoroborate Salts of the Cis-chlorobis-(triethylphosphine)(3-trifluoromethyl, 5-methyl-pyrazole) platinum(II) and cis-chlorobis-(triethylphosphine)(indazole) platinum(II) cations". *Inorg. Chem.,* 22, 774, 1983.

Peer Reviewed Papers -- Continued

19. (C) Atwood, J.L.; Zaworotko, M.J. "The Formation and Structure of the Novel Aluminoxane Anion $[Me_2AlOAlMe_3]_2^{2-}$". *J. Chem. Soc., Chem. Comm.,* 302, 1983.

20 (C) Bushnell, G.W.; Fjeldsted, D.O.K; Stobart, S.R.; Zaworotko, M.J. "Two-Centre Oxidative Addition of Hexafluorobut-2-yne to a Bis(μ-pyrazolyl)-di-iridium(I) Complex Leading to Bridge Elimination via H-transfer from Coordinated COD: X-ray Crystal Structure of a Mixed-bridge Mixed-Valence Iridium Dimer Incorporating a (1-3,5,6 η-C_8H_{11}) Ligand." *J. Chem. Soc., Chem. Comm.,* 580, 1983.

21. (N) Atwood, J.L.; Berry, D.E.; Stobart, S.R.; Zaworotko, M.J. "Aspects of Organocadmium Chemistry. Part 3. Cyclometallated Alkyls and Aryls of Zn, Cd and Hg and the Crystal and Molecular Structure of Bis[o-N,N-dimethyl-amino-methyl)phenyl]-mercury(II)." *Inorg. Chem.,* 22, 3480, 1983.

22. (F) Beveridge, K.A.; Bushnell, G.W.; Stobart, S.R.; Atwood, J.L.; Zaworotko, M.J. "Pyrazolyl-bridged Iridium Dimers. 4. Crystal and Molecular Structures of Bis(cycloocta-1,5-diene) bis(μ-pyrazolyl) diiridium(I), its Dirhodium(I) Isomorph, and Two Bis(cycloocta-1,5-diene) Analogues Incorporating 3,5-disubstituted μ-pyrazolyl Ligands." *Organometallics,* 2, 1447, 1983.

23. (C) Auburn, M.J.; Holmes-Smith, R.D.; Stobart, S.R.; Zaworotko, M.J.; Cameron T.S.; Kumari, A. "The Phosphinomethylsilyl Group as a Bifunctional Bridging Ligand. X-ray Crystal Structure of Hexacarbonyl bis(μ-diphenylphosphino-methyldimethylsilyl)diruthenium(II), and of its Reaction Product with Trifluoroacetic Acid, a Mononuclear Ruthenium(II) Complex Incorporating a Unique Co-ordinated Silanol." *J. Chem. Soc., Chem. Comm.,* 1523, 1983.

24. (C) Decker, M.J.; Fjeldsted, D.O.K.; Stobart, S.R.; Zaworotko, M.J. "Weak Intermetallic Bonding. A Rare Example of Molecular Stacking in a Neutral Square-planar Second-row Transition Metal Complex. X-ray Crystal Structures of [Rh(cod)(C1)(dmpH)] and $[Rh(CO)_2](C1)(pzH)]$ (cod = cycloocta-1,5-diene; dmpH = 3,5-dimethylpyrazole; pzH = pyrazole)." *J. Chem. Soc., Chem. Comm.,* 1525, 1983.

25. (C) Bushnell, G.W.; Stobart, S.R.; Vefghi, R.; Zaworotko, M.J. "Addition of Diphenylphosphine to a Bis(μ-pyrazolyl)diiridium(I) Complex Resulting in H-transfer to a Co-ordinated Cycloocta-1,5-diene: X-ray Crystal Structure of an Iridium Dimer Incorporating Unsymmetrical Pyrazole and Phosphido Bridging Groups and 1-,4-5-η-C_8H_{13} Ligand." *J. Chem. Soc. Chem. Comm.,* 282, 1984.

26. (C) Stobart, S.R. and Zaworotko, M.J. "Influence on Transition-metal Arene Complex Formation of Hydrogenation and Rearrangement of Polyaromatic Substrates Induced by Aluminum Trichloride. Octahydrophenanthrene Complexes from Tetralin: X-ray Crystal and Molecular Structure of η^5-1,2,3,4,5,6,7,8,9-endo-nonahydro-9-exo-methylphenanthrenyl(tri-carbonyl) manganese." *J. Chem. Soc., Chem. Comm.,* 1700, 1984.

Peer Reviewed Papers -- Continued

27. (F) Atwood, J.L.; Beveridge, K.A.; Bushnell, G.W.; Dixon, K.R.; Eadie, D.T.; Stobart, S.R.; Zaworotko, M.J. "Pyrazolyl-bridged Iridium Dimers. 6. Two-fragment, Two-center Oxidative Addition of Halogens and Methyl Iodides to trans-bis(triphenylphosphine)dicarbonyl bis(μ-pyrazolyl)diridium(I)." *Inorg. Chem.,* 23, 4050, 1984.

28. (C) Auburn, M.J.; Grundy, S.L.; Stobart, S.R.; Zaworotko, M.J. "Phosphinoalkylsilyl Complexes. 6. Isolation of a Silyl Complex of Iridium(I). Crystal and Molecular Structure of Dicarbonyl-(triphenylphosphine)[(diphenylphosphinoethyl)dimethylsilyl]iridium." *J. Amer. Chem. Soc.,* 107, 266, 1985.

29. (F) Zaworotko, M.J.; Kerr, C.R.; Atwood, J.L. "Reaction of the Phenoxide ion with Trimethylaluminum. Isolation and Crystal Structure of [K.dibenzo-18-crown-6][Al_2Me_6OPh] and K[$AlMe_2(OPh)_2$]." *Organometallics,* 4, 238, 1985.

30. (F) Bushnell, G.W.; Fjeldsted, D.O.K.; Stobart, S.R..; Zaworotko, M.J.; Knox, S.A.R.; MacPherson, K.A. "Pyrazolyl-bridged Iridium Dimers. 7. Synthesis and Properties of Bridge-substituted Analogues of [$Ir(COD)(\mu\text{-}pz)]_2$, pzH = pyrazole, the 'Mixed Bridge' Complex [$Ir_2(COD)_2(\mu\text{-}pz)(\mu fpz)$], pfzH-3,5,-bis(trifluro-methyl)pyrazole, and the 'Mixed Metal' Dimer [$IrRh(COD)_2(\mu pz)_2$]. Crystal and Molecular Structures of Bis(cyclooctadiene)-bis(μ-3-phenyl-5-methylpyrazolyl) diiridium(I) (Disymmetric Isomer) and Bis(cycloocta-1,5-diene)bis-(μ-3,4,5-trimethylpyrazolyl) diiridium(I)". *Organometallics,* 4, 1107, 1985.

31. (F) Zaworotko, M.J.; Stamps, R.J.; Ledet, M.T.; Zhang, H; Atwood, J.L. "Heterocyclophane Complexes of Transition Metals. 1. Synthesis and Crystal Structure of Both (η^5-[2,2](2,5)pyrolloparacyclophane) tricarbonylchromium and ((η^5[2,2](2,5)pyrolloparacyclophane) tricarbonylchromium." *Organometallics,* 4, 1697, 1985.

32. (C) Fjeldsted, D.O.K.; Stobart, S.R.; Zaworotko, M.J. "Pyrazolyl-bridged Iridium Dimers. 10. Sequential Addition at the Metal Centers in a Diiridium Configuration. Oxidatively Induced Relocation of a Bent, Terminal Nitrosyl Group to Occupy a Bridging Site." *J. Amer. Chem. Soc.,* 107, 8258, 1985.

33. (C) Stobart, S.R.; Zaworotko, M.J. "Hydrogenation and Rearrangement of Coal Related Aromatic and Hydroaromatic Molecules Promoted under Mild Conditions by Aluminum Trichloride." *Fuel,* 64, 1623, 1985.

34. (F) Hood, R.D.; Vedel-MacRander, G.C; Zaworotko, M.J.; Tatum, F.M.; Meeks, R.G. "Distribution, Metabolism, and Fetal Uptake of Pentavalent Arsenic in Pregnant Mice Following Oral and Intraperitoneal Administration." *Teratology,* 35, 19, 1987.

Peer Reviewed Papers -- Continued

35. (F) Holmes-Smith, R.D.; Stobart, S.R.: Vefghi, R.; Zaworotko, M.J.; Jochem, K.; Cameron, T.S. "Phosphinoalkylsilyl Complexes, Part 7." *J. Chem. Soc. Dalton Trans.,* 969, 1987.

36. (F) Hood, R.D., Vedel, G.C.; Zaworotko, M.J.; Tatum, F.M.; Meeks, R.G. "Uptake, Distribution and Metabolism of Trivalent Arsenic in the Pregnant Mouse." *J. Tox. Environ. Health,* 25, 419, 1988.

37. (C) Gaudet, M.V.; Peterson, D.C.; Zaworotko, M.J. "Ternary Hydrogen Halide/base/benzene Mixtures: A New Generation of Liquid Clathrates." *J. Incl. Phenom.,* 6, 425, 1988.

38. (C) Cameron, T.S.; Clerk, M.D.; Linden, A; Sturge, K.C.; Zaworotko, M.J. "Interaction of Trialkylaluminum Reagents with Organotransition Metal -complexes: One Step, High Yield Ethylation of $[(arene)_2Fe]^{2+}$ cations." *Organometallics,* 7, 2571, 1988.

39. (F) Gaudet, M.V.; Hanson, A.W.; White, P.S.; Zaworotko, M.J. "Synthetic Applications of Trialkyaluminum Reagents: Alkylation and Reduction of Transition Metal Complexed π-hydrocarbon Ligands." *Organometallics,* 8, 286, 1989.

40. (F) Dubois, R.H.; Zaworotko, M.J.; White, P.S. "Conformational Variability in π-Complexes of Hexaethylbenzene. Crystal and Molecular Structure of $[C_6Et_6)(C_5H_5)Fe]BPh_4$." *J. Organomet Chem.,* 362, 155, 1989.

41. (N) Gaudet, M.V.; Zaworotko, M.J.; White, P.S. "X-ray Crystallographic Characterization of Hydrogen Bonding Interactions in Piperidinium Chloride and Piperidinium Tetrachloraluminate: Relevance to the Structure of Room Temperature Melts." *Inorg. Chem.,* 28, 1191, 1989.

42. (F) Gaudet, M.V.; Zaworotko, M.J.; Cameron, T.S.; Linden, A. "Crystal and Molecular Structure and Unsual Hydrocarbon Solubility Properties of $(C_5H_5)_2ZrCl(AlC1_4)$: Relevance to Ziegler-Natta Polymerization Chemistry." *J. Organomet. Chem.,* 367, 267, 1989.

43. (C) Dubois, R.H.; White, P.S.; Zaworotko, M.J. "Complex Hydrogen Bonded Cations. X-ray Crystal Structure of $[(benzylammonium)_4C1][A1C1_4]_3$ and its Relevance to the Structure of Basic Chloroaluminate Room Temperature Melts." *Inorg. Chem.,* 28, 2019, 1989.

44. (C) Clerk, M.D.; Sturge, K.C.; White, P.S.; Zaworotko, M.J. "Trimethylaluminum Mediated Chloromethylation of $([arene]_2Fe)^{2+}$ Cations." *J. Organomet. Chem.,* 368, C33, 1989.

45. (F) Zaworotko, M.J.; Cameron, T.S.; Linden, A.; Sturge, K.C. "Structures of the Tetrachloroaluminate Salts of the N-ethylpyridinium, 2-ethylpyridinium, Pyridinium and 1-chloromethyl 1,2,3,4,5,6-hexamethylbenzenonium Cations." *Acta Cryst.,* C45, 996, 1990.

Peer Reviewed Papers -- Continued

46. (F) Zaworotko, M.J.; Sturge, K.C.; White, P.S. "Crystal and Molecular Structure of (^{5}-1,3,5-trimethyl-6-phenylcyclohexadienyl)(η^5-cyclopentadienyl)iron, a Mixed Full Sandwich Iron(II) Complex." J. Organomet. Chem., 389, 333, 1990.

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23. Zaworotko, M.J. **Keynote lecture**, "Aspects of Noncovalent Bonding", 1995 Atlantic CIC Student Conference, Halifax, May 1995.
24. Zaworotko, M.J. **Invited lecture**, "Molecular Recognition in Solids: Cocrystals by Design", 78th Canadian Chemical Conference, May 1995, Guelph, Ontario.
25. Zaworotko, M.J. **Invited lecture**, "From Molecules to Crystals", NATO ARW on Modular Chemistry, September 1995, Estes Park, Colorado, USA.
26. Zaworotko, M.J. **Invited lecture**, "Crystal Engineering of Functional Solids", ESTAC annual meeting, November 1995, Toronto, Canada.
27. Zaworotko, M.J. **Invited poster**, "Non-interpenetrated Molecular Ladders", NATO ARW on Modular Synthesis, May 1996, Montreal, Canada.
28. Zaworotko, M.J. **Invited lecture**, "Chains, Planes and Frames: Crystal Engineering of Transition Metal Sustained Coordination Polymers", 5th International Summer School on Supramolecular Chemistry, June 1996, Ustron Poland.
29. Zaworotko, M.J. **Invited microsymposium lecture,** "Chains, Planes and Frames: How the Dimensionality of Hydrogen Bonded or Coordination Polymer Networks Influences Crystal Morphology", XVII International Union of Crystallography Congress, August 1996, Seattle, USA.

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34. Zaworotko, M.J. **Invited Lecture**, "Polymorphism in Network Polymers". 17th European Crystallographic Meeting, August 1997, Lisbon, Portugal.
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47. Zaworotko, M.J. **Invited Lecture**, "Zeolite and Clay Mimics: Design Strategies for Generation of Organic and Metal-Organic Adsorbents". Gordon Conference on Zeolites and Layered Materials, June 1999.
48. Zaworotko, M.J. **Invited Lecture**, "From Molecules to Crystals". Fourth International Conference on Materials Chemistry, Dublin, Ireland, July 1999.
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50. Zaworotko, M.J. **Invited Lecture**, "Novel Technology for Hog Manure Odour Control/Remediation". HEMS annual symposium, Ottawa, Canada, December 1999.
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53. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of Zeolite and Clay Mimics". 83rd Canadian Society for Chemistry Conference, Calgary, Alberta, May 2000.
54. Zaworotko, M.J. "From Molecules to Crystals… and Back Again". 13th International Symposium on Surfactants in Solution, Gainesville, Florida, June 2000.
55. Zaworotko, M.J. **Invited Lecture**, "Design and Function of 2D and 3D Coordination Polymers". Royal Society of Chemistry, Dalton Discussion on Inorganic Crystal Engineering, Bologna, Italy, September 2000.
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59. Zaworotko, M.J. **Invited Lecture**, "Coexisting Covalent and Noncovalent Nets". ACS SW/SE Regional Meeting, New Orleans, Louisiana, December 2000.
60. Zaworotko, M.J. **Invited Lecture**, "Composite Materials by Design". Pacifichem 2000, Hawaii, December 2000.
61. Zaworotko, M.J. **Invited Lecture**, "Self-assembly of Discrete and Infinite Nanoscale Structures". AMRI-DARPA, New Orleans, February 2001.
62. Zaworotko, M.J. **Invited Lecture**, "Self-assembly of Discrete and Infinite Nanoscale Structures". XV International School-Seminar Spectroscopy of Molecular and Crystals, Chernihiv, Ukraine, June 2001.
63. Zaworotko, M.J. **Invited Lecture**, "Crystals and Nanocrystals by Design". ECM 20, "Supramolecular Materials Microsymposium", Krakow, Poland, August 2001.
64. Zaworotko, M.J. **Invited Lecture**, VIII International Seminar on Inclusion Compounds, Poland, September 2001.
65. Zaworotko, M.J. **Invited Lecture**, International Symposium on Crystal Chemistry, Chisinau, Moldova, October 2001.
66. Zaworotko, M.J. **Invited Lecture**, "From Molecules to Crystals: Crystal Engineering of Network Solids", 59th Pittsburgh Diffraction Conference, Cincinnati, October 2001.
67. Zaworotko, M.J. **Invited Lecture**, Modern Trends in Inorganic Chemistry, Calcutta, India, December 2001 (cancelled because of 9/11 related issues).
68. Zaworotko, M.J. **Invited Lecture**, Singapore International Chemical Conference, Singapore, December 2001 (cancelled because of 9/11 related issues).

Conference Presentations (excluding presentations by co-workers) – Cont'd

69. Zaworotko, M.J. **Invited Keynote Lecture**, Research Trends in Science and Technology 2002, Beirut and Byblos, Lebanon, March 2002.
70. Zaworotko, M.J. **Invited Lecture**, "Finite and Infinite Polygonal Assemblies", 223rd American Chemical Society National Meeting, Orlando, April 2002.
71. Zaworotko, M.J. **Invited Lecture**, "Binary Crystals by Design", 223rd American Chemical Society National Meeting, Orlando, April 2002.
72. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering with Pharmaceuticals: Design of the Composition and Structure of Pharmaceutical Phases", Higuchi Research Seminar, Kansas, May 2002.
73. Zaworotko, M.J. **Invited Lecture**, "Self-assembly of Crystals and Nanocrystals", American Crystallographic Association, San Antonio, May 2002.
74. Zaworotko, M.J. **Invited Lecture**, "Self-assembly of Crystals and Molecules with Nanoscale Features". 85th Canadian Society for Chemistry Conference, Vancouver, British Columbia, June 2002.
75. Zaworotko, M.J. **Invited Microsymposium Lecture**, 19th Congress and General Assembly of the International Union of Crystallography, Geneva, Switzerland, August 2002.
76. Zaworotko, M.J. **Invited Lecture**, SE Region ACS, Charleston, November 2002.
77. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of the Composition of Pharmaceutical Phases", ACS Prospective on Polymorphism in Crystals, Tampa, February 2003.
78. Zaworotko, M.J. "Crystal Engineering of the Composition of Pharmaceutical Solids", 225th ACS National Meeting, New Orleans, March 2003.
79. Zaworotko, M.J.; Moulton, B.; Lu, J.; McManus, G.; Wong, R.; Rather, B. **Invited Lecture**, "Crystal Engineering of Coordination Compounds with Nanoscale Features", FAME 2003, Orlando, May 2003.
80. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of the Composition of Pharmaceutical Phases", Strategies for Improving Solubility Workshop, Philadelphia, June 2003.
81. Zaworotko, M.J. **Invited Lecture**, "From Crystal Engineering of Coordination Polymers to Design of Nanoscale Molecules", 39th IUPAC Congress, Ottawa, Canada, August 2003.
82. Zaworotko, M.J. **Invited Lecture**, "Coordination Polymers with Nanoscale Features", 39th IUPAC Congress, Ottawa, Canada, August 2003.
83. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of the Composition of Pharmaceutical Phases", 39th IUPAC Congress, Ottawa, Canada, August 2003.
84. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of the Composition of Pharmaceutical Phases", Strategies for Improving Solubility Workshop, Brussels, Belgium Oct. 2003.
85. Zaworotko, M.J. **Invited Lecture.** "Self-Assembly of Nanoscale Chemical Structures", ICMAT, Singapore, Dec. 2003.
86. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of the Composition of API's ", Determining Factors for Measuring Permeability Workshop, Philadelphia, January 2004.
87. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of the Composition of Pharmaceutical Phases", ACS Prospective on Polymorphism in Crystals, Tampa, February 2004.
88. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of the Composition of API's", Polymorphism and Crystallization Forum 2004, Princeton, April 2004.

Conference Presentations (excluding presentations by co-workers) – Cont'd

89. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Coordination Compounds with Nanoscale Features", 80th FAME 2004, Orlando, May 2004.
90. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of the Composition of Pharmaceutical Phases", Solubility Workshop, Philadelphia, June 2004.
91. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of the Composition of API's", Polymorphism Workshop, Philadelphia, June 2004.
92. Zaworotko, M.J. **Invited Lecture.** "Supramolecular Synthesis", Green Chemistry Gordon Conference, July 2004.
93. Zaworotko, M.J. **Invited Lecture.** "Do Pharmaceutical Co-crystals Represent a Path to Improved Medicines", Strategies for Improving Solubility Workshop, Brussels, Belgium Sep. 2004.
94. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of the Composition of API's", Polymorphism, Crystallization and Salt Selection Workshop, Washington, DC, February 2005.
95. Zaworotko, M.J. **Invited Lecture.** "Pharmaceutical Co-crystals – Do They Represent a New Path to Improved Medicines", Polymorphism and Crystallization 2005, London, UK, March 2005.
96. Zaworotko, M.J. **Invited Lecture.** "Pharmaceutical Co-crystals – Do They Represent a New Path to Improved Medicines", American Crystallographic Association Annual Meeting, Orlando, May 2005.
97. Zaworotko, M.J. **Invited Lecture.** "From Molecules to Crystals – and Back Again", American Crystallographic Association Annual Meeting, Orlando, May 2005.
98. Zaworotko, M.J. **Invited Lecture.** "The role of co-crystals in supramolecular and solid-state chemistry", 2nd International Conference on Green and Sustainable Chemistry and 9th Annual Green Chemistry and Engineering Conference, Washington DC, June 2005.
99. Zaworotko, M.J. **Invited Lecture.** "From Molecules to Crystals – and Back Again", Rare Earth Research Conference (RERC05), Keystone, Colorado, June 2005.
100. Zaworotko, M.J. **Invited Microsymposium Lecture**, Polymorphism in Co-crystals and Pharmaceutical Co-crystals", 20th Congress and General Assembly of the International Union of Crystallography, Florence, Italy, August 2005.
101. Zaworotko, M.J. **Invited Lecture.** "From molecular building blocks to binary and ternary nets", European Science Foundation meeting on "Supramolecular Chemistry: Molecular Architectures and Systems", Obernai, France, October 2005.
102. Zaworotko, M.J. **Keynote Lecture.** "From Molecules to Crystals… and Back Again", Singapore International Chemistry Conference IV, Singapore, December 2005.
103. Zaworotko, M.J. **Invited Lecture.** Pacifichem 2005, Honolulu, Hawaii, December 2005.
104. Zaworotko, M.J. **Invited Lecture.** "Realizing the potential of co-crystals as novel patentable materials", Devloping IP strategies for crystalline forms conference, London, UK, December 2005.
105. Zaworotko, M.J. **Invited Lecture.** "Designing Pharmaceutical Co-crystals", Polymorphism and Crystallization 2006, London, UK, March 2006.
106. Zaworotko, M.J. **Invited Lecture.** "Design and preparation of co-crystals", ACS Prospective on Process Crystallization in the Pharmaceutical and Chemical Industry, Philadelphia, April 2006.
107. Zaworotko, M.J. **Invited Lecture.** "Do pharmaceutical co-crystals represent a new path to improved medicines?", FAME2006, Orlando, May 2006.
108. Zaworotko, M.J. **Invited Lecture.** "Crystal engineering of metal-organic nets with form and function", FAME2006, Orlando, May 2006.

Conference Presentations (excluding presentations by co-workers) – Cont'd

109. Zaworotko, M.J. **Invited Lecture.** "The Role of Co-crystals in Pharmaceutical Form and Formulation", Polymorphism and Crystallization 2006, IQPC Meeting, San Diego, May 2006.
110. Zaworotko, M.J. **Invited Lecture.** 89th Canadian Society for Chemistry Conference, Halifax, Nova Scotia, May 2006.
111. Zaworotko, M.J. **Invited Lecture.** "Design and preparation of co-crystals", INDABA5, Kruger National Park, South Africa, August 2006.
112. Zaworotko, M.J. **Invited Lecture.** "Pharmaceutical co-crystals: do they represent a new path to improved medicines?", British Pharmaceutical Conference, Manchester, UK, September 2006.
113. Zaworotko, M.J. **Invited Lecture.** "Polymorphism in co-crystals", Pharmaceutical co-crystals, IQPC Meeting, Amsterdam, September 2006.
114. Zaworotko, M.J. **Invited Lecture.** "The role of co-crystals in supramolecular and solid-state chemistry", XVth Conference on Physical Methods in Coordination and Supramolecular Chemistry, Chisinau, Moldova, September 2006.
115. Zaworotko, M.J. **Invited Lecture.** "Design and preparation of co-crystals", ACS Prospective on Crystallization Process Development: Case Studies & Research, Boston, February 2007.
116. Zaworotko, M.J. **Opening Plenary Lecture.** XIth International Seminar on Inclusion Compounds, Kyiv, Ukraine, June 2007.
117. Zaworotko, M.J. **Invited Lecture.** "Back to the future for metal-organic materials", Nano-Structured Porous Materials Workshop, DOD meeting, Washington DC, September 2007.
118. Zaworotko, M.J. **Invited Lecture.** "New Intellectual Property Opportunities for Old Natural Products via New Crystal Forms", Pharmaceutical Co-Crystals 2007, IQPC Meeting, Amsterdam, September 2007.
119. Zaworotko, M.J. **Invited Lecture.** SERMACS 2007, Greenville, SC, October 2007.
120. Zaworotko, M.J. **Plenary Lecture.** "Back to the future for metal-organic materials", 5th National Symposium on Structural Chemistry (5th NSSC), China and the Symposium on Chinese Strategy of Crystal Growth & Design, Fujian, China, October 2007.
121. Zaworotko, M.J. **Invited Lecture.** Polymorphism and Crystallization Conference, Clearwater, FL, November 2007.
122. Zaworotko, M.J. **Invited Lecture.** "Co-crystals by design: New opportunities for old natural products through crystal engineering." Polymorphism and Crystallization, Philadelphia, December 2007.
123. Zaworotko, M.J. **Invited Lecture.** "The role of co-crystals in pharmaceutical science", International Symposium on "Challenges and Innovations in Pharmaceutical Research" State University of Morelos, Cuernavaca, Mexico, March 2008.
124. Zaworotko, M.J. **Invited Lecture.** "Co-crystals involving chiral co-crystal formers", Polymorphism and Crystallization 2008, London, UK, March 2008.
125. Zaworotko, M.J. **Invited Lecture.** "Back to the future: new ligands for old topologies". 235th ACS National Meeting, New Orleans, LA, April 2008.
126. Zaworotko, M.J. **Invited Lecture.** "Increasing solubility by crystal engineering and co-crystal formation". 35th Annual Meeting & Exposition of the Controlled Release Society, New York, July 2008.
127. Zaworotko, M.J. **Invited Lecture.** "Pharmaceutical co-crystals."236th ACS National Meeting, Philadelphia, PA, August 2008.

Conference Presentations (excluding presentations by co-workers) – Cont'd

128. Zaworotko, M.J. **Invited Keynote Lecture.** "The role of co-crystals in pharmaceutical science." 21st Congress and General Assembly of the International Union of Crystallography, Osaka, Japan, August 2008.
129. Zaworotko, M.J. **Keynote Speaker.** "New approaches to solvent-free synthesis: co-crystal controlled solid-state synthesis (C^3S^3)", British Pharmaceutical Congress, Manchester, September 2008.
130. Zaworotko, M.J. **Invited Lecture.** "The broader opportunities for co-crystals in drug discovery and development", Pharmaceutical Cocrystals 2008, Amsterdam, September 2008.
131. Zaworotko, M.J. **Invited Lecture.** "Pharmaceutical Co-crystals: History and Relevance to Drug Development", Drug Formulation 2008, Philadelphia, November 2008.
132. Zaworotko, M.J. "Crystal engineering strategies for improving the bioavailability of low solubility drugs", UK-US International Alzheimer's Disease Symposium, Tampa, November 2008.
133. Zaworotko, M.J. **Invited Lecture**, "The broader opportunities for co-crystals in drug discovery and development", Indo-US Bilateral Workshop on Cocrystals and Polymorphs, Mysore, India, February 2009.
134. Zaworotko, **Invited Lecture**, "From molecules to crystals, and back again", 38th National Seminar on Crystallography, Mysore, India, February 2009.
135. Zaworotko, M.J. **Invited Lecture**, "Pharmaceutical co-crystals – Do they represent multiple paths to new and improved medicines", Latin American Symposium on Polymorphism and Crystallization in Drugs and Medicines, Sao Pedro, Brazil, March 2009.
136. Zaworotko, M.J. **Plenary Lecture**, "From Molecules to Crystals and Back Again: Crystal Engineering of Functional Solids", Taibah International Chemistry Conference, Al Madinah, Saudi Arabia, March 2009.
137. Zaworotko, M.J. **Invited Lecture**, "The Role of Cocrystals in Pharmaceutical Science and Solid-State Chemistry", Molecules, Materials, Medicine, M^3-2009, Santa Barbara, May 2009.
138. Zaworotko, M.J. **Three Invited Lectures**, 2009 Beijing Summer School on "Crystallography, Crystal Engineering and Functional Materials", Beijing, China, July 2009.
139. Zaworotko, M.J. **Invited Lecture**, "Achieving Greater Product Differentiation through Pre-Formulation Technologies", Product Enhancement Technologies in Pharmaceutical Life-Cycle Management, Philadelphia, September 2009.
140. Zaworotko, M.J. **Invited Lecture**, "Structure-Property Relationships in Multiple Component Crystals", Pharmaceutical Co-Crystals 2009, Amsterdam, September 2009.
141. Zaworotko, M.J. **Keynote Lecture**, "From molecules to crystals and metal-organic materials: What have we learned from the past 20 years?", 2nd Asian Conference on Coordination Chemistry, Nanjing, China, November 2009.
142. Zaworotko, M.J. **Invited Lecture**, "Making the right haystacks for the right needles: Metal-organic platforms from discrete metal-organic containers", 239th ACS National Meeting, San Francisco, CA, March 2010.
143. Zaworotko, M.J. **Invited Lecture**, "The role of co-crystals in green chemistry: Solvent-free synthesis of new ligands", FAME2010, Palm Harbor, Florida, May 2010.
144. Zaworotko, M.J. **Invited Lecture**, "Hierarchy of supramolecular synthons and their role in selection of cocrystal former libraries", 5th Bologna Convention on Crystal Forms, Bologna, Italy, September 2010.

Conference Presentations (excluding presentations by co-workers) – Cont'd

145. Zaworotko, M.J. **Invited Lecture**, "Advances in synthesis of pharmaceutical cocrystals", Association for Crystallization Technology 2010, Rutgers University, NJ, October 2010.
146. Zaworotko, M.J. **Invited Lecture**, "Pharmaceutical Cocrystals: A new path to improved medicines", 9th Polymorphism & Crystallization Scientific Forum, Philadelphia, October 2010.
147. Zaworotko, M.J. **Invited Lecture**, "New ligands for discrete and infinite metal-organic materials", Pacifichem 2010. Honolulu, Hawaii, December 2010.
148. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering: Where have we come from and where do we go next?", Current Trends in Crystal Engineering Research Symposium, Indian Institute of Science, Bangalore, India, December 2010.
149. Zaworotko, M.J. **Plenary Lecture**, "Form Before Function: Why Design Still Matters to Crystal Engineering", International Symposium on Facets of Weak Interactions in Chemistry, Kolkatta, India, January 2011.
150. Zaworotko, M.J. **Invited Lecture**, "Metal-organic material platforms for small molecule separations and ion exchange", 241st ACS National Meeting, Anaheim, CA, March 2011.
151. Zaworotko, M.J. **Invited Lecture**, Crystal engineering of properties in metal-organic and molecular solids", XXth International Conference on the Chemistry of the Organic Solid-State, Bangalore, India, June 2011.
152. Zaworotko, M.J. **Invited Lecture**, "Form before function: Why design still matters to crystal engineering", 242nd ACS National Meeting, Denver, CO, August 2011.
153. Zaworotko, M.J. **Invited Lecture**, "Form still comes before function: Why design still matters to crystal engineering", 13th International Seminar on Inclusion Compounds, Gierloz, Poland, September 2011.
154. Zaworotko, M.J. **Invited Lecture**, "Ensuring successful crystal engineering of multi-component crystals", Pharmaceutical Co-Crystals 2011, Amsterdam, September 2011.
155. Zaworotko, M.J. **Keynote Lecture**, "Crystal Engineering of Organic and Metal-Organic Materials", NanoFlorida 2011, Miami, FL, October 2011.
156. Zaworotko, M.J. **Invited Lecture**, "Form before function: Why design still matters to crystal engineering", Garnett E. Peck Symposium on Pharmaceutical Solids, Purdue University, October 2011.
157. Zaworotko, M.J. **Keynote Address**, "Form before function: Why design still matters to crystal engineering", 35th Senior Technical Meeting of the Puerto Rico Section of the American Chemical Society, Dorado, PR, November 2011.
158. Zaworotko, M.J. **Invited Lecture**, "Crystal engineering of multiple component crystalline solids", CF@Bo Meeting, Bologna, Italy, January 2012.
159. Zaworotko, M.J. **Invited Lecture**, "Crystal engineering of multi-component crystals: Why there is still room for design", Indo-US meeting on the Evolving Role of Solid-State Chemistry in Pharmaceutical Science, Manesar, near Delhi, India, February 2012.
160. Zaworotko, M.J. **Invited Lecture**, "Why crystal engineering means that there is still plenty of room at the bottom", KAUST Advanced Membranes and Porous Materials Center, Inaugural Symposium, March 2012.

Conference Presentations (excluding presentations by co-workers) – Cont'd

161. Zaworotko, M.J. **Invited Lecture**, "Selective gas adsorption of carbon dioxide by metal-organic material platforms", 243rd ACS National Meeting, San Diego, CA, March 2012.
162. Zaworotko, M.J. **Invited Lecture**, "Why topology matters to crystal engineers", Beautiful Crystals for the World – A Celebration of Michael O'Keeffe's Half a Century of Contributions to Symmetry and Patterns in Chemistry", Swanage, UK. May 2012.
163. Zaworotko, M.J. **Invited Lecture**, "The role of crystal engineering and cocrystals in pharmaceutical science", Symposium on Applications of Solid state Physics to Pharmaceuticals, Brazilian Physics Society, Aguas de Lindoia, Brasil, May 2012.
164. Zaworotko, M.J. **Invited Lecture**, "The crystal engineering approach to development of cocrystal former libraries", M3 - Molecular, Materials, Medicines, Banff, Canada May 2012.
165. Zaworotko, M.J. **Invited Lecture**, "Crystal engineering of multicomponent materials: Why there is still plenty of room at the very bottom", 244th ACS National Meeting, Philadelphia, Pennsylvania, August 2012.
166. Zaworotko, M.J. **Invited Lecture**, "How pore size control affects carbon dioxide uptake in porous materials", 244th ACS National Meeting, Philadelphia, Pennsylvania, August 2012.
167. Zaworotko, M.J. **Invited Lecture**, "Co-crystal former libraries and solubility enhancement", Pharmaceutical Co-Crystals 2012, Amsterdam, September 2012.
168. Zaworotko, M.J. **Keynote Lecture**, "Crystal engineering of task specific metal-organic materials", 6th National Symposium on Structural Chemistry, Suzhou, China, October 2012.
169. Zaworotko, M.J. **Invited Lecture**, "Smartly Designed Materials", Chemistry: Synthesis, Structure and Dynamics. A Conference on Crystal Engineering organized by the Indian Institute of Science and University of Hyderabad, Orange County Resort, Coorg, India, December 2012.
170. Zaworotko, M.J. **Invited Lecture**, "Product Diversity and Enhancement through Pre-Formulation Technologies with an Emphasis on Co-crystal Technology", 2nd Enhancing Drug bioavailability of Solubility Conference, Boston, MA, January 2013.
171. Zaworotko, M.J. **Invited Lecture**, "Catalytically active metal-organic materials", 245th ACS National Meeting, New Orleans, Louisiana, April 2013.
172. Zaworotko, M.J. **Invited Lecture**, "Metal-organic materials design in the context of carbon dioxide separations", 245th ACS National Meeting, New Orleans, Louisiana, April 2013.
173. Zaworotko, M.J. **Invited Lecture**, "Smart(ly Designed) Materials", Frontiers in Condensed Matter Sciences, Fortaleza, Brasil, April 2013.
174. Zaworotko, M.J. **Invited Lecture**, "Smart(ly Designed) Materials", Showcase symposium of FAME 2013, Innisbrook, Florida, May 2013.
175. Zaworotko, M.J. **Plenary Lecture**, "Smart(ly Designed) Materials", Past, Present and Future of Crystallography: From Small Molecules to Macromolecules and Supramolecular Structures, Milan, Italy, June 2013.
176. Zaworotko, M.J. **Invited Lecture**, "Smart(ly Designed) Materials", 7th CF@Bo Meeting, Bologna, Italy, June 2013.
176. Zaworotko, M.J. **Invited Lecture**, "Why Topology Matters to Crystal Engineers", SIAM Mathermatical Aspects of Materials Science 2013, Philadelphia, June 2013.
177. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of Task-Specific Porous Materials", ICMAT 2013, Singapore, July 2013.

Conference Presentations (excluding presentations by co-workers) – Cont'd

178. Zaworotko, M.J. **Plenary Lecture**, "Crystal engineering of porous material platforms", Asian Crystallographic Association Meeting, Hong Kong, December 2013.
179. Zaworotko, M.J. **Invited Lecture**, "Crystal engineering of metal-organic material (MOM) platforms", International Symposium on MOF and Related Open Framework Materials, Macao, December 2013.
180. Zaworotko, M.J. **Invited Lecture**, "Putting the squeeze on CO_2 with narrow pore metal-organic materials", Symposium I, Materials for Carbon Capture, Materials Research Society, Spring Meeting, San Francisco, April 2014.
181. Zaworotko, M.J. **Invited Lecture**, "Multi-Component Pharmaceutical Materials", 11th International Conference & Exhibition on Polymorphism and Crystallization, Orlando, FL, May 2014.
182. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of Task-Specific Materials", 1st International Symposium on Halogen Bonding, Porto Cesareo, Italy, June 2014.
183. Zaworotko, M.J. **Keynote Lecture**, "Crystal Engineering of Task-Specific Materials", Materials and Surface Science Institute Annual Meeting, Limerick, June 2014.
184. Zaworotko, M.J. **Plenary Lecture**, "Crystal Engineering of porous metal-organic materials", 13th International Symposium on Advancing the Chemical Sciences, Dublin, Ireland, July 2014.
185. Zaworotko, M.J. **Invited Lecture**, "Hybrid Organic-Inorganic Materials for Carbon Capture", Metal-Organic Frameworks: Experiments and Simulations, Telluride Science Research Center, July 2014.
186. Zaworotko, M.J. **Plenary Speaker**, "Crystal Engineering of Task-Specific Materials", University of Malaya Pharmaceutical Co-Crystal Symposium 2014, Kuala Lumpur, July 2014.
187. Zaworotko, M.J.; Schoedel, A. S.; Elsaidi, S. **Invited Lecture**. "Crystal engineering of new metal-organic material platforms", 41st International Conference on Coordination Chemistry, Singapore, July 2014.
188. Zaworotko, M.J. **Invited Lecture**, "From crystal structures to crystal engineering – 102 years of x-ray diffraction and still going strong". Boston Biotech Symposium 2014, Boston August 2014.
189. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering of Metal-Organic Platforms with Inorganic Anions", 248th ACS National Meeting, SanFrancisco, California, August 2014.
190. Zaworotko, M.J. **Keynote Lecture**, "From crystal structures to crystal engineering – 102 years of x-ray diffraction and still going strong", The Institute of Chemistry of Ireland Congress 2014, September 2014.
191. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of Porous Materials", MOF2014, Kobe, Japan, September 2014.
192. Zaworotko, M.J. **Invited Lecture**, "Crystal Engineering of Task-Specific Materials", ACS Mid-West Regional Meeting, Columbia, Missouri, November 2014.
193. Zaworotko, M.J. **Invited Lecture**, "The Impact of Pharmaceutical Cocrystals upon API Solubility and Bioavailability", Solubility and Bioavailability Summit 2014, Philadelphia, PA, December 2014.
194. Zaworotko, M.J.; Lusi, M.; Perry, M., **Invited Lecture**, "Pharmaceutical Cocrystals", Polymorphism in Pharmaceutical Solids, SSPC Course for Industry, Limerick, Ireland, January 2015.
195. Zaworotko, M.J. **Invited Lecture**. "How crystals are made and why this really matters", 1st Brasil-Ireland Science Week, Dublin, Ireland, February 2015.

Conference Presentations (excluding presentations by co-workers) – Cont'd

196. Zaworotko, M.J. **Invited Lecture**. "A general perspective on crystal engineering", CECAM workshop on Industrial Challenges of Crystallization, Nucleation and Solubility: Perspectives from Industry, Experiment and simulation, UCD, Dublin, Ireland, June 2015.
197. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Porous Materials", 98th Canadian Chemistry Conference and Exhibition, Ottawa, Canada, June 2015.
198. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Porous Materials", ICMAT2015, Singapore, June 2015.
199. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials", CASE2015, Dublin, June 2015.
200. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Porous Materials", MC12, 12th International Conference on Materials Chemistry, York, England, July 2015.
201. Zaworotko, M.J. **Commemorative Speaker.** "Hybrid Porous Materials", Golden Jubilee Chemistry Conference, Singapore, August 2015 (declined).
202. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Porous Materials", Gordon Research Conference on Nanoporous Materials and Their Applications, Holderness, NH, August 2015.
203. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Porous Materials", ISIC-15, 15th International Seminar on Inclusion Compounds, Warsaw, Poland, August 2015.
204. Zaworotko, M.J. **Invited Lecture.** "Hybrid Porous Materials", ECM29, 29th European Crystallography Meeting, Rovinj, Croatia, August 2015.
205. Zaworotko, M.J. **Plenary Lecture**. "Crystal Engineering of Task-Specific Materials", 1st Latin American Crystallography Association Meeting, Sao Paulo, Brasil, September 2015.
206. Zaworotko, M.J. **Invited Lecture.** "High Pressure Applications of Porous Metal-Organic Materials", 2015 IUCr High Pressure Workshop, Campinas, Brasil, September 2015.
207. Zaworotko, M.J. **Invited Lecture.** "Why Crystals Matter to the Real World", Science Week 2015, Limerick, November 2015.
208. Zaworotko, M.J. **Eva Philbin Award Lecture.** "Crystal Engineering of Task-Specific Materials – Pharmaceutical Materials", University College Dublin, November 2015.
209. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials", Pacifichem 2015, Honolulu, December 2015.
210. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering: Form to Function", Fundamentals of the Solid Form: New Insights and Developments, RSC Burlington House, London, March 2016.
211. Zaworotko, M.J. **Keynote Lecture.** "Crystal Engineering of Hybrid Ultramicroporus Materials", 1st Middle-Eastern Materials Science Conference, Abu Dhabi, March 2016.
212. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering: Form to Function", British Crystallographic Association Spring Meeting, Nottingham, UK, April 2016.
213. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering: Form to Function", Science Atlantic Chemistry Conference, Halifax, Canada, June 2016.
214. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering of Hybrid Ultramicroporous Materials", 99th Canadian Chemistry Conference and Exhibition, Halifax, Canada, June 2016.
215. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering: Its Relevance to Pharmaceutical Science," 3rd Annual Advanced API Convention, Mumbai, India, July 2016.

Conference Presentations (excluding presentations by co-workers) – Cont'd

216. Zaworotko, M.J. **Keynote Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials", MOF 2016, Long Beach, CA, USA, September 2016.
217. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Ultramicroporus Materials", 3rd ICSU/IUPAC Workshop on Crystal Engineering, Milan, Italy, Feb 24th 2017.
218. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering of Multi-Component Pharmaceutical Materials", 23rd International Conference on the Chemistry of the Organic Solid State, Stellenbosch, South Africa, April 2017.
219. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Porous Materials", North America-Greece-Cyprus Workshop on Paramagnetic Materials, Paphlos, Cyprus, May 2017 (cancelled).
220. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials", 67th meeting of the American Crystallographic Association, New Orleans, USA, May 2017.
221. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials", 100th meeting of the Canadian Society for Chemistry, Toronto, Canada, June 2017.
222. Zaworotko, M.J. **Invited Lecture.** "Are Hydrates the Nemesis of Crystal Engineering." 9th CF@Bo Meeting, Bologna, Italy, June 2017.
223. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering: Form to Function." 20th International Symposium on Industrial Crystallization, Dublin, Ireland, September 2017.
224. Zaworotko, M.J. **Keynote Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials." 2nd International Conference on Advanced Energy Materials, AEM2017, University of Surrey, UK, September 2017.
225. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering and its Relevance to Pharmaceutical Science." Workshop titled "Active Pharmaceutical Ingredients: Rational Approach to Process Development", APIS Chem S.r.l., Milan, Italy, September 2017.
226. Zaworotko, M.J. **Invited Lecture.** "Why Crystals Matter to the Real World." 36th ChemEd-Ireland Conference, Limerick, Ireland, October 2017.
227. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Task-Specific Materials." British Crystallographic Association Joint Industrial/Chemical Group 2017 Autumn Meeting, Cambridge, UK, November 2017.
228. Zaworotko, M.J. **Invited Lecture.** "Why Crystals Matter to the Real World." BioPharma Ambition Conference, Dublin, Ireland, February 2018.
229. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering of Task-Specific Materials." Symposium on Inorganic Materials, Galway, Ireland, March 2018.
230. Zaworotko, M.J. **Keynote Lecture.** "Why Crystals Matter to the Real World." J&J Engineering Showcase 2018, Limerick, Ireland, May 2018.
231. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering of Task-Specific Materials." International School of Crystallization, Granada, Spain, May 2018.
232. Zaworotko, M.J. **Keynote Lecture.** "Taxonomy of Porous Solids: A Crystal Engineering Perspective." 2nd Meeting on Porous Molecular Solids, Vietri sul Mare, Italy, June 2018.
233. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering of Task-Specific Materials." Crystallize COST Action, Annual Meeting, Cork, Ireland, May 2018.
234. Zaworotko, M.J. **Invited Lecture.** "Crystal Engineering Then and Now." Gordon Research Conference on Crystal Engineering, Newry, ME. USA, June 2018.

Conference Presentations (excluding presentations by co-workers) – Cont'd

235. Zaworotko, M.J. **Keynote Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials." 43rd International Conference on Coordination Chemistry, Sendai, Japan, 1st August 2018.
236. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering of Hybrid Ultramicroporous Materials." 1st European Symposium on Sorption Science, Vienna, Austria, 6th September 2018.
237. Zaworotko, M.J. **Plenary Lecture.** "Crystal Engineering: Then and Now." 9th International Conference on Materials Science and Condensed Matter Physics, Chisinau, Moldova, 27th September 2018.
238. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then and Now." 1st China-Ireland Symposium on Cocrystals and Crystal Engineering, Tianjin, China, 23rd October 2018.
239. Zaworotko, M.J. **Public Lecture.** "Why Crystals and Crystal Engineering Matter to the Real World." Royal Society of Chemistry, Burlington House, London, UK, 29th November 2018.
240. Zaworotko, M.J. **Plenary Lecture**. "Crystal Engineering: Then and Now." Symposium on Oral Solid Dosage Forms, Gothenberg, Sweden, 8th April 2019.
241. Zaworotko, M.J. **Keynote Lecture.** "Crystal Engineering: Then and Now." 37th Biennial Meeting of the Royal Spanish Society of Chemistry, San Sebastian, Spain, May 29th, 2019.
242. Zaworotko, M.J. **Keynote Lecture.** "Crystal Engineering: Then and Now." 14th International Symposium on Macrocyclic and Supramolecular Chemistry, Lecce, Italy, 4th June 2019.
243. Zaworotko, M.J. **Invited Lecture.** "Stimuli Responsive Crystals." 10th CF@Bo Meeting, Bologna, Italy, 9th June 2019.
244. Zaworotko, M.J. **Magisterial Lecture.** "Crystal Engineering: Custom Design of the Right Material for the Right Application." 25th International Federation of Societies of Cosmetic Chemists, Milan, Italy, 30th September 2019.
245. Zaworotko, M.J. **Invited Lecture**. "Why Crystal Engineering will Save the World: Disruptive Technologies to Enable Energy Sustainability", Irish Development Authority Annual Meeting, 6th January 2020.
246. Zaworotko, M.J. **Keynote Lecture**. "Why Crystals will Save the World" BCA/BCAG Online Joint Spring Meeting 2021 (remote lecture), 31st March 2021.
247. Zaworotko, M.J. **Spiers Memorial Lecture.** "Why Crystals (including MOFs) will Save the World", Faraday Discussion on the theme of "MOFs for energy and the environment" (remote lecture), 23rd June 2021.
248. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering of Ultramicroporous Materials", 25th Congress of the International Union of Crystallography, Prague (remote lecture), 15th August 2021.
249. Zaworotko, M.J. **Invited Lecture**. "The Chemistree of Porous Solids", XXIX International Materials Research Congress, Cancun, Mexico, 16th August 2021.
250. Zaworotko, M.J. **Plenary Lecture**. "The Chemistree of Porous Solids", 22nd YUCOMAT (remote lecture), 1st September 2021.
251. Zaworotko, M.J. **Invited Lecture**. "Cocrystals: Then and Now", 11th CF@Bo Meeting, Bologna, Italy (remote lecture), 11th September 2021.
252. Zaworotko, M.J. **Keynote Lecture**. "Why Crystals will Save the World", International Conference on Sustainable Energy-Water-Environment Nexus in Desert Climate 2021 (remote lecture), 23rd November 2021.

Conference Presentations (excluding presentations by co-workers) – Cont'd

253. Zaworotko, M.J. **Invited Lecture**. "Crystal engineering of Ultramicroporous Materials", Pacifichem 2021 (remote lecture), 18th December 2021.
254. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then and Now". North America Greece Cyprus Meeting 2022, Ayia Napa, Cyprus, 10th May 2022.
255. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then and Now". 1st School of Supramolecular and Bionanomaterials, Lake Como, Italy, 16th June 2022.
256. Zaworotko, M.J. **Invited Webinar**. "Why Crystals will Save the World", *Crystal Growth & Design* webinar series (remote lecture), 20th July 2022.
257. Zaworotko, M.J. **Plenary Lecture**. "Why Crystals will Save the World", Sixth Silk Road International Expo, Xi'an, China (remote lecture), 16th August 2022.
258. Zaworotko, M.J. **Keynote Lecture**. "The "Chemistree" of Porous Solids", EuChemS 2022 Meeting, Lisbon, Portugal, August 31st 2022.
259. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then and Now", Mid-West Region ACS Meeting, Iowa City, USA, October 20th 2022.
260. Zaworotko, M.J. **Invited Webinar**. "Why Crystals will Save the World", Micromeritics Webinar Series, 2nd November 2022.
261. Zaworotko, M.J. **Invited Lecture.** "Why Crystals will Save the World", Institute of Ireland Centenary Congress, Dublin, Ireland, November 17th 2022.
262. Zaworotko, M.J. **Keynote Lecture**. "Crystal Engineering: Then and Now", Dutch Association for Crystal Growth, 50th Anniversary Symposium, Amsterdam, The Netherlands, 20th March 2023.
263. Zaworotko, M.J. **Invited Lecture**. "The "Chemistree" of Porous Solids", ACS National Meeting, Indianapolis USA, 26th March 2023.
264. Zaworotko, M.J. **Plenary Lecture**. "Crystal Engineering: Then and Now", 1st MASA Materials Science Conference, Skopje, Macedonia, 27th May 2023.
265. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then, Now and Next", Telluride Workshop on Porous Materials, Telluride, CO, USA, 11th June 2023.
266. Zaworotko, M.J. **Invited Lecture**. "Cocrystals: Then, Now and Next", 12th CF@Bo Meeting, Bologna, Italy, 12th September 2023.
267. Zaworotko, M.J. **Invited Lecture**. "Materials for a Sustainable World", Nobel Symposia 193 Satellite Meeting, Stockholm, Sweden, 18th September 2023.
268. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then, Now and Next", Nobel Symposia 193, Karlskoga, Sweden, 19th September 2023.
269. Zaworotko, M.J. **Plenary Lecture**. "Crystal Engineering: Then, Now and Next", 2nd Texas Pore Engineering Conference, Denton, TX, 22nd October 2023.
270. Zaworotko, M.J. **Plenary Lecture**. "Why Crystals will Save the World", Science Week 2024, UM6P, Benguerir, Morocco. 13th February 2024.
271. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then, Now and Next", 9th EuChemS Meeting, Dublin, Ireland, 9th July 2024.
272. Zaworotko, M.J. **Keynote Lecture**. "Crystal Engineering of Ultramicroporous and Nonporous Physisorbents", MOF2024, Singapore, 16th July 2024.
273. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then, Now and Next", 4th IUPAC Workshop on Crystal Engineering, Milan, 26th August 2024.

Conference Presentations (excluding presentations by co-workers) – Cont'd

274. Zaworotko, M.J. **Invited Lecture**. "Polymorphism in Coordination Networks and Molecular Solids. ECM34, Padova, Italy, 30th August 2024.
275. Zaworotko, M.J. **Invited Lecture**. "Crystal Engineering: Then, Now and Next", 17th ISIC Meeting, Poznan, Poland, 5th September 2024.
276. Zaworotko, M.J. **Invited Lecture**. "Materials for a Sustainable World", XXI Brasilian Meeting on Inorganic Chemistry, Belo Horizonte, Brasil, 14th September 2024.
277. Zaworotko, M.J. **Plenary Lecture**. "Why Crystals Will Save The World", VI Latin American Crystallographic Association Meeting, Montevideo, Uruguay, 24th September 2024.
278. Zaworotko, M.J. **Invited Lecture**. "Why Crystals Will Save The World", CELTIC 2024 China Meeting, Xi'an, China, 3rd November 2024.

Conference/symposium organizer

1. ACS Great Lakes Regional Meeting, DeKalb Illinois, 1990. A symposium on organometallics in synthesis.
2. Halifax CSC 1991: a symposium devoted to main group chemistry.
3. Halifax Atlantic CIC: conference titled "*Synthetic Chemistry in Atlantic Canada*"
4. Guelph CSC 1995: symposium titled "*Back to the Future: A Symposium Celebrating 100 years of X-rays*".
5. Newfoundland CSC 1996: symposium titled "*Extended Metal Systems*" (with C.R. Lucas).
6. Nova Scotia, NATO ASI 1996: two week summer school titled "*Crystal Engineering: The Design and Application of Functional Solids*" (with K.R. Seddon).
7. Cancun, 5th N. American Chemical Congress 1997. "*Crystal Engineering*" (with R.D. Rogers).
8. Washington DC American Crystallographic Association Meeting 1998, Transactions Symposium. "*Crystal Engineering*" (with R.D. Rogers).
9. Anaheim ACS Meeting 1999: Inorganic Division symposium "*Transition metal coordination polymers*" (with R.D. Rogers)
10. Glasgow IUCr, 1999: symposium devoted to crystal engineering (with R.D. Rogers, G.R. Desiraju)
11. Gordon Research Conference, 2000, "*Organic Structures and Properties*" (co-chair with M.D. Ward)
12. Pacifichem 2000, Hawaii, 2000, symposium devoted to applications of crystal engineering (with R.D. Rogers)
13. SERMACS 2002, Charleston, SC, "*Crystals and Nanocrystals by Design*" (with W.T. Pennington).
14. 2nd International Conference on Green and Sustainable Chemistry and 9th Annual Green Chemistry and Engineering Conference, Washington DC, June 2005, "*Non-covalent Derivatization*" (with W. Jones).
15. Indo-USF forum on "*The evolving role of solid-state chemistry in pharmaceutical science*", Delhi, India, February 2012 (with G.R. Desiraju).
16. 2nd Gordon Research Conference on "*Crystal Engineering*", June 2012 (co-vice-chair with C. Aakeröy).
17. 3rd Gordon Research Conference on "*Crystal Engineering*", June 2014 (co-chair with C. Aakeröy).

PhD Students Graduated

Student Name	Current Position
1. K. Craig Sturge	Industry, Canada (Pharma)
2. Brian Moulton	Industry, USA (Software)
3. Zhenqiang Wang	University of South Dakota, USA (Faculty)
4. Elisabeth Rather	Industry, USA (Legal)
5. Heba Abourahma	New Jersey College, USA (Faculty)
6. Gregory McManus	Florida Gulf Coast University, USA (Faculty)
7. Tanise Shattock	Industry, USA (Pharma)
8. Joanna Bis	Industry, USA (Pharma)
9. David Weyna	Industry, USA (Pharma)
10. John J. Perry IV	unknown
11. Miranda Perry (nee Cheney)	Industry, USA (Pharma)
12. Padmini Kavuru	Industry, USA (Pharma)
13. Jason Perman	Industry, USA (Materials)
14. Heather Clarke	Industry, USA (Pharma)
15. ZhenJie Zhang	Nankai University, China (Faculty)
16. Alexander Schoedel	Florida Institute of Technology, USA (Faculty)
17. Zhuxiu Zhang	Nanjing Tech University, China (Faculty)
18. Sameh Elsaidi	Illinois Institute of Technology, USA (Faculty)
19. Mona Mohamed	Illinois Institute of Technology, USA (Faculty)
20. Tien Teng Ong	Industry, Singapore (Pharma)
21. Naga Duggirala	Industry, USA (Pharma)
22. Daniel O'Nolan	Industry, USA (Materials)
23. Amrit Kumar	Industry, Ireland (Materials)
24. Krishna Peraka	Industry, Ireland (Pharma)
25. Sarah Foley	Industry, Ireland (Materials)
26. Rana Sanii	Industry, Ireland (Pharma)
27. ShiYuan Zhang	Zhejiang University, China (Faculty)
28. Mohana Shivanna	Sandia National Laboratory, USA (Post-Doc)
29. ShiQiang Wang	ICES, Singapore (Research staff)
30. Naveen Kumar	Industry, UK (Materials)
31. Molly Haskins	Industry, Ireland (Pharma)
32. Daniel O'Hearn	Industry, Canada (Pharma)
33. Meiyan Gao	University of California, Berkeley (Post-Doc)
34. ShaSha Jin	Industry, China (Pharma)
35. Li Xia	University of Limerick (Post-Doc)
36. Deng Chenghua	University of Chicago (Post-Doc)
37. Maryam Rahmani	Industry, Ireland (Materials)
38. Yassin Andaloussi	Industry, UK (Materials)
39. Aizhamal Subanbekova	University of Limerick (Post-Doc)
40. Kyriaki Koupepidou	Kyoto University (Post-Doc)